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# **TRANSACTIONS OF THE AMERICAN • SOCIETY • FOR METALS**



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**Number 4**

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# *The* TRANSACTIONS *of the* AMERICAN SOCIETY FOR METALS

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RAY T. BAYLESS, *Editor*

Vol. XXVI

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No. 4

*The object of the Society shall be to promote the arts and sciences connected with either the manufacture or treatments of metals, or both.*

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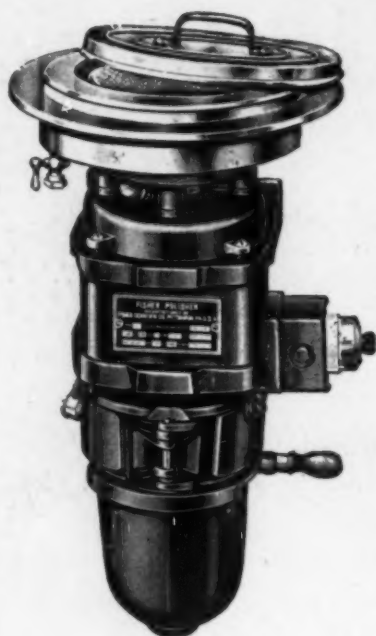
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## Table of Contents

A Study of Deoxidation Type Inclusions in Alloy Steels—	
By W. A. Hare and Gilbert Soler .....	903
Discussion .....	926
Some Problems in the Production of Low Carbon Sheets in Non-Continuous	
Mills—By M. L. Samuels and Alfred Boyles .....	929
Discussion .....	946
Relation of Size of Spheroids in Tool Steel to Its Machinability and to Holding	
Edge of Cutter—By Donald E. Roda .....	950
Discussion .....	958
Recovery of Cold-Worked Nickel on Annealing—By Erich Fetz .....	961
Discussion .....	987
The Effects of Longitudinal Scratches on Valve Spring Wire—	
By F. P. Zimmerli, W. P. Wood and G. D. Wilson .....	997
Discussion .....	1017
Metal Coloring—By C. B. F. Young .....	1019
Discussion .....	1031
A New Application for the Short-Time High Temperature Tensile Test—	
By C. L. Clark, A. E. White and G. J. Guarnieri .....	1035
Discussion .....	1049
The Effect of Grain Size on the Oxidation of a Low Carbon Steel—	
By C. A. Siebert and Clair Upthegrove .....	1051
Discussion .....	1059
Frictional Wear Tests of Lubricated Surfaces—	
By C. J. Coberly and Fred M. Arnold .....	1064
Some Unusual Aspects of Malleable Iron Melting—By Alfred L. Boegehold ....	1084
Taylor Speed and Its Relation to Reduction of Area and Brinell Hardness—	
By E. J. Janitzky .....	1122
Technical Program and Report of Officers, A.S.M.—20th Annual Convention,	
Detroit, October 17 to 21, 1938 .....	1132
Annual Address of the President .....	1135
Report of the Treasurer .....	1137
Annual Report of the Secretary .....	1141
Election of Officers .....	1149
Annual Banquet .....	1150
The Scientific Approach to our Problems—By Benjamin F. Fairless .....	1154



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## A STUDY OF DEOXIDATION TYPE INCLUSIONS IN ALLOY STEELS

### Their Nature, Properties and Relation to Furnace Practice

BY WESTON A. HARE AND GILBERT SOLER

#### Abstract

*This paper summarizes the results of an extensive series of tests on deoxidation type inclusions as they occur in alloy steels and discusses the formation and effect of various types of inclusions as they occur in commercial steels.*

*Inclusions were separated from the steel by chemical means. After separation, metallographic, petrographic, X-ray and chemical analyses and studies were made, determining the mineralogical phases of various types of nonmetallic inclusions in several types of steel. The relation existing between the known inclusion phases and equilibrium diagrams of the oxides is discussed.*

*The effect of variations in furnace practice on inclusions is discussed in some detail and a table presented showing the chemical analysis of the inclusions, their mineralogical phases, the deoxidation practice used in making the steel and the per cent FeO in the steel before the final deoxidation was effected as determined by analysis of the gases evolved from the molten metal.*

**B**EARING parts, which are particularly subject to fatigue stresses, have always required an exceptionally clean grade of steel. It is to be expected, therefore, that this company should exert every effort to reduce the inclusion content of its steels to the minimum. Research has played an important part in this practical development and considerable progress has been made in the manufacture of clean steel by suitable control of melting and deoxidation practices.

Every effort is being made to eliminate definitely harmful inclusions and to reduce the nonmetallic inclusions to the point where they will have no deleterious influence on the finished product. However, the fact that a certain nonmetallic oxide content is necessarily present in any killed steel must be accepted and due allowance made

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. Of the authors, Weston A. Hare is research engineer and Gilbert Soler is manager of research, Steel and Tube Division, Timken Roller Bearing Co., Canton, Ohio. Manuscript received June 25, 1937.



for it. In fact, certain types of inclusions may be produced deliberately with beneficial results.

Granting therefore that a certain amount of deoxidation type inclusions will always be present in alloy steels, the problem is to control the manufacturing process in such a manner as to assure that the user will have a material suited to his particular needs. In some cases certain types of inclusions are not objectionable even in relatively large quantities. In other cases the quantity must be held to a minimum. In still other instances, for best results it is essential that the type of inclusion be controlled. Consequently, we are endeavoring to determine what type, size and shape of inclusion is least detrimental so that we can manufacture steel accordingly.

During the past two years we have made a special study of the nature and distribution of nonmetallic deoxidation type inclusions as they occur in alloy steels, correlating these data with changes in melting practice and the properties of the steels. This work involved separating inclusions of various types, analyzing them by chemical, petrographic and X-ray methods as well as by the usual metallographic methods. Much work still remains to be done, but important data on the effect of inclusions as well as their nature and amount under various practices have been obtained.

#### EFFECT OF INCLUSIONS IN STEEL

It appears that the nature of the inclusions in steel is even more important than the quantity. Inclusions whose softening point is low tend to elongate during rolling, forming stringers. Likewise, stringers may be formed by the aggregation of large numbers of fine particles of high melting point oxides in the ingot, the area elongating to some extent during rolling.

However, if the inclusions are uniformly distributed through the steel and have a softening point above the rolling temperature, no elongation will occur and consequently a larger percentage by weight can be tolerated for certain purposes.

The degree of elongation of inclusions during rolling depends upon the nature of the individual included particles, rather than on the composite analysis of all the inclusions present. Consequently, our work involved a study of the individual types of inclusions and their combinations under given conditions as well as a metallurgical study of their distribution in the sample.



## METHODS OF STUDYING INCLUSIONS

There are four general methods of studying nonmetallic inclusions, each having certain advantages. By combining these several methods a complete knowledge of the inclusions present, their properties, and some information as to their origin and therefore control, may be secured.

The usual test, or inspection, for inclusions is by means of a metallographic microscope. Here such inclusions as are exposed on a polished surface may be partially identified by their visual appearance, or less often by their reaction to selective etchants. This is a rapid method and has the advantage of showing the inclusions "in situ," which allows the arrangement of the particles to be determined. That feature is of particular importance, as frequently the arrangement of inclusions is more important than the type or amount.

However, the field of observation is extremely limited and a little calculation will show that it would take several hundred metallographic observations to equal in volume of metal studied a single 20-gram chemical analysis. Consequently, the problem arises as to how to secure a fair sample or how many observations are required to gain an accurate and fair knowledge of the amount and type of nonmetallic inclusions present. Therefore, if the speed of metallographic inspection is to be utilized and only one or even a few samples observed, a sufficiently wide range of acceptance should be established to balance the possibility of a single specimen being substandard.

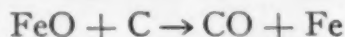
Suitable chemical treatment permits the investigator to separate the inclusions from the steel and determine their chemical composition and quantity.

Petrographic analysis of the inclusions separated from the steel by chemical means enables the actual phases or combinations of oxides representing each type of nonmetallic inclusion to be determined. This study may in turn be supplemented by X-ray technique. The data presented in this paper were secured by using these several methods, modified where necessary to meet the special problems presented in alloy steels.

## METHOD OF GAS ANALYSIS

This method consists of drawing a sample of molten steel from a sampling spoon into a specially designed evacuated tube and of

analyzing the evolved gases volumetrically. This not only measures the gases as such evolved from molten steel, but has also been shown to measure with satisfactory accuracy the FeO present in the steel sample. The dissolved FeO reacts with dissolved carbon as follows:



and inasmuch as the reaction has been shown to go largely to com-

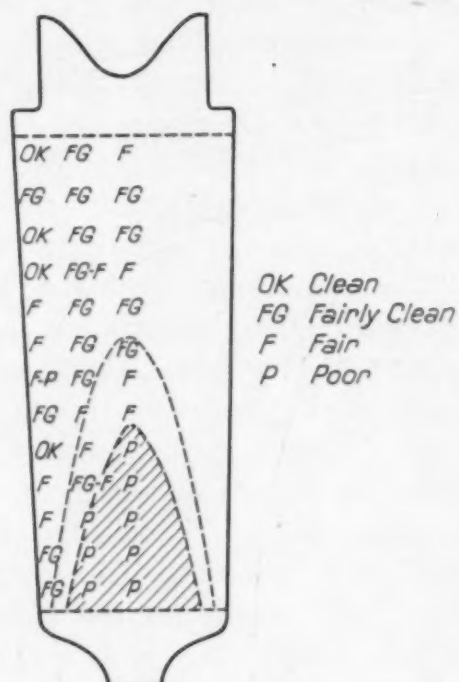


Fig. 1—Cross Section of Typical Ingot. Rated in 4-Inch Square for Cleanliness at Points Shown.

pletion under the conditions of the test, the weight percentage of carbon monoxide obtained may be calculated to per cent FeO.<sup>1</sup>

#### EXPERIMENTAL PROCEDURE

Typical segregation of inclusion material in an ingot as determined by metallographic inspection is shown in Fig. 1. To assure representative specimens in the work under discussion, solid samples consisting of either entire cross section slices of rolled bars or "pie slice" sections were used, depending upon the material being studied. All were taken from "middles" of the ingot product. As previous work has shown that drillings were subject to error due to the high

<sup>1</sup>W. Hare, L. Peterson and G. Soler, "An Improved Method for Determining Gas Content of Molten Steels," TRANSACTIONS, American Society for Metals, Vol. 25, 1937, p. 889.

oxide content of fine drillings as compared to coarse, all samples were cut with a saw and filed with a clean file prior to analysis. The very close checks obtained on duplicate runs of the same heat indicate that the samples used were representative and satisfactory.

In order to secure satisfactory material for petrographic analysis, we utilized the method for separating inclusions developed by F. W. Scott. This method employs an electrolytic decomposition of the

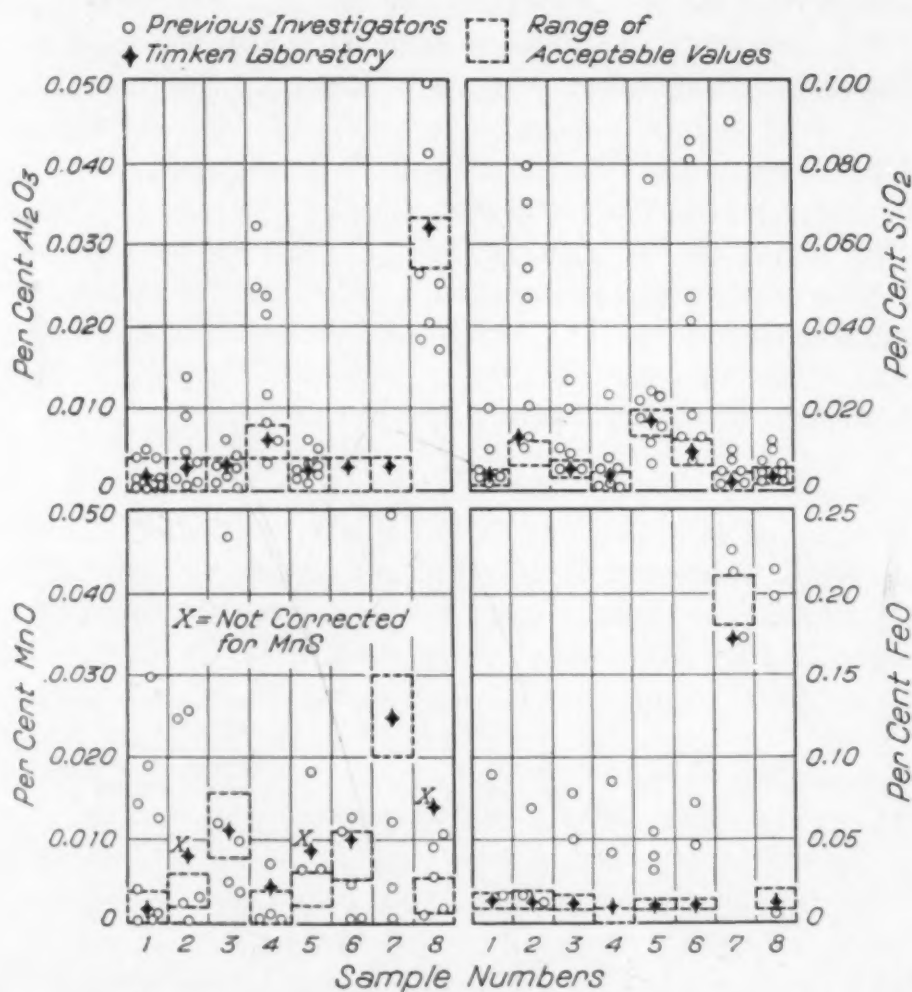


Fig. 2—Oxides Present in Bureau of Standards Samples as Determined Electrically by Various Laboratories.

steel sample as the anode in an electrolyte of magnesium iodide, with subsequent purification of the residue by chemical means. In our work we carried out the electrolytic decomposition in a neutral atmosphere to prevent oxidation of the iron salts. This modified method has been described elsewhere in detail.<sup>2</sup>

<sup>2</sup>W. A. Hare and Gilbert Soler, *Metals and Alloys*, Vol. 8, 1937, p. 169-172.



**Applicability**—Plain carbon steels, nickel steels, nickel molybdenum and carbon molybdenum steels, and the low carbon nickel chromium steels may be decomposed satisfactorily by the electrolytic method referred to above. However, medium carbon nickel-chromium steel contains too much carbon and chromium and the residue is contaminated by chromium carbide. Likewise, high carbon chromium steels cannot be studied satisfactorily by this method, but require dry decomposition by means of chlorine gas.

**Accuracy**—Scott has shown that the electrolyte used in the separation does not affect  $\text{Al}_2\text{O}_3$  or  $\text{SiO}_2$  nor silicates unless the  $\text{FeO}$  or  $\text{MnO}$  content is above 60 per cent. We have checked all the washing reagents used for  $\text{MnO}$  and  $\text{FeO}$  solubility. To further check our work we have analyzed the eight Bureau of Standards samples used in the "Cooperative Study"<sup>3</sup> and find, as shown in Fig. 2, that our oxide analyses were well within their "acceptable" range in practically every case. There is some probability, however, that the values for per cent  $\text{FeO}$  may be somewhat high, due to a slight contamination of the residue during separation.

## RESULTS

The set of data shown in Table I summarize the results obtained in this work. The heats listed, while not necessarily typical of their specifications, were chosen to give a wide range of inclusion contents and are listed in order of increasing ratio of  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  content.

Table I												
Sample	Steel Type	Analysis of Inclusions Per Cent by Weight				Mineralogical Phase Estimated Per Cent Present						
		Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	MnO	FeO	1	2	3	4	5	6	7
A	1015	0.0049	0.0123	0.0036	0.0092	5	90	..	..	..	..	5
B	4615	0.0082	0.0061	0.0030	0.0109	..	..	80	..	..	10	10
C	4615	0.0107	0.0047	0.0033	0.0120	..	..	..	90	..	..	10
D	4620	0.0102	0.0031	0.0042	0.0112	10	15	10	..	..	..	65
E	1065 + Mo	0.0085	0.0023	0.0038	0.0127	15	..	..	..	25	..	60
F	4615	0.0163	0.0031	0.0029	0.0095	10	..	5	..	..	85	..
G	1065 + Mo	0.0159	0.0020	0.0043	0.0145	5	..	60	..	10	25	..
H	1065 + Mo	0.0210	0.0026	0.0036	0.0133	10	..	..	..	..	90	..

The phases listed in Table I are described in the following paragraphs:

Phase 1. Quartz ( $\text{SiO}_2$ ). This occurs as fairly large roughly broken pieces probably of exogenous nature, possibly washed into the steel from fireclay refractories, this material being composed of grains of quartz embedded in a colloidal clay matrix.

<sup>3</sup>J. G. Thompson, H. C. Vacher and H. A. Bright, American Institute of Mining and Metallurgical Engineers, Technical Publication No. 758, 1936.



Phase 2. A glass of about 1.59 refractive index, yellow in appearance, found in inclusions of lower  $\text{Al}_2\text{O}_3$  content. This phase probably contains considerable FeO and MnO which raise its refractive index.

Phase 3. A glass of refractive index of 1.50 to 1.56. This is thought to be an aluminum silicate glass, probably containing a little iron and manganese oxide. Probably the refractive index increases with the amount of  $\text{Al}_2\text{O}_3$  present.

Phase 4. A glass of refractive index 1.53 containing mullite crystals and thought to be of fireclay origin. This was found in one or two samples only.

Phase 5. A glass of 1.65-1.66 refractive index of conchoidal fracture. This phase often contained corundum particles, so is probably high in alumina content.

Phase 6. Round or nearly round aggregations of nearly pure corundum ( $\text{Al}_2\text{O}_3$ ) probably containing a small amount of mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) within them. The clusters are obviously made up of a number of small individual particles of corundum sintered together to form a larger mass. A micrograph of this type inclusion (635X), Fig. 11, shows this structure clearly.

Phase 7. Pure corundum ( $\text{Al}_2\text{O}_3$ ) present in individual particles, not coalesced or sintered together as in Phase 6.

These phases are listed in order of decreasing  $\text{SiO}_2$  and increasing  $\text{Al}_2\text{O}_3$  content. The correlation of the chemical analysis of the inclusions with the mineralogical phases found petrographically will be noted in the table.

Photomicrographs of both a polished metallographic section (above) and of the separated inclusions in the petrographic sample (below) are shown at 200 diameters for each heat listed in the table in the accompanying figures and are discussed in order.

It will be noted in Fig. 3a that the inclusions shown in the metallographic section are composed entirely of elongated stringers, rated as low melting point silicates. Corresponding to these, the separated inclusions in Fig. 3b, are seen to be composed of broken fragments of glass, Phase 2, mechanically clumped together in the petrographic photomicrograph. The nature of this glass may be deduced from the chemical analysis to be largely  $\text{SiO}_2$  with lesser amounts of  $\text{Al}_2\text{O}_3$ , MnO and FeO.

Fig. 4 shows a heat of silicon and aluminum-killed steel of greater  $\text{Al}_2\text{O}_3$  content. The metallographic photomicrograph shows

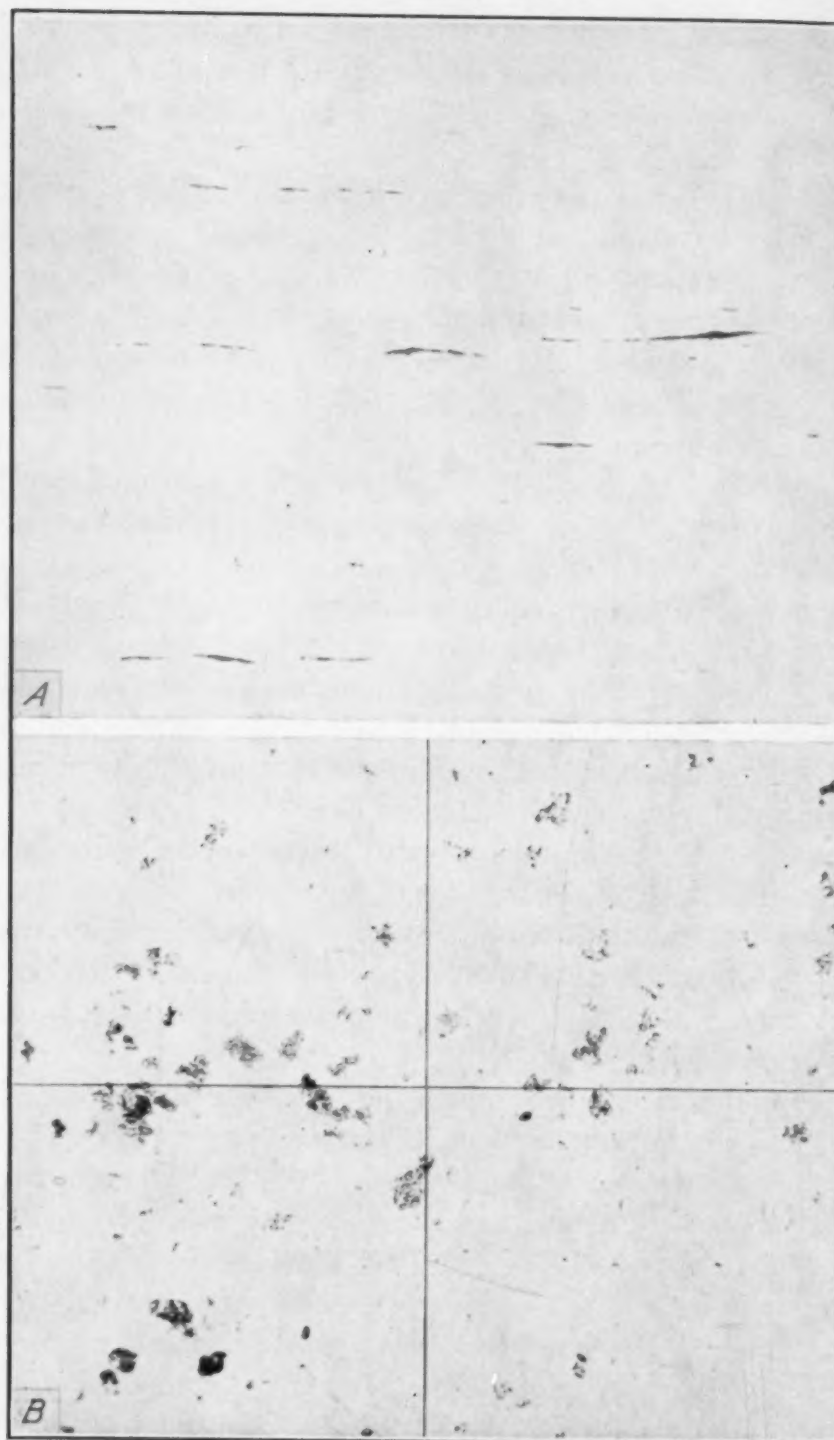


Fig. 3—Metallographic and Petrographic Photographs of Sample A.  $\times 200$ . (A) Metallographic Sample. (B) Petrographic Sample.

several roughly round  $\text{Al}_2\text{O}_3$  inclusions and a moderately elongated silicate stringer. In the petrographic photomicrograph Fig. 4b, the round bright particles (out of focus) and other rounded particles of

$\text{Al}_2\text{O}_3$   
visib

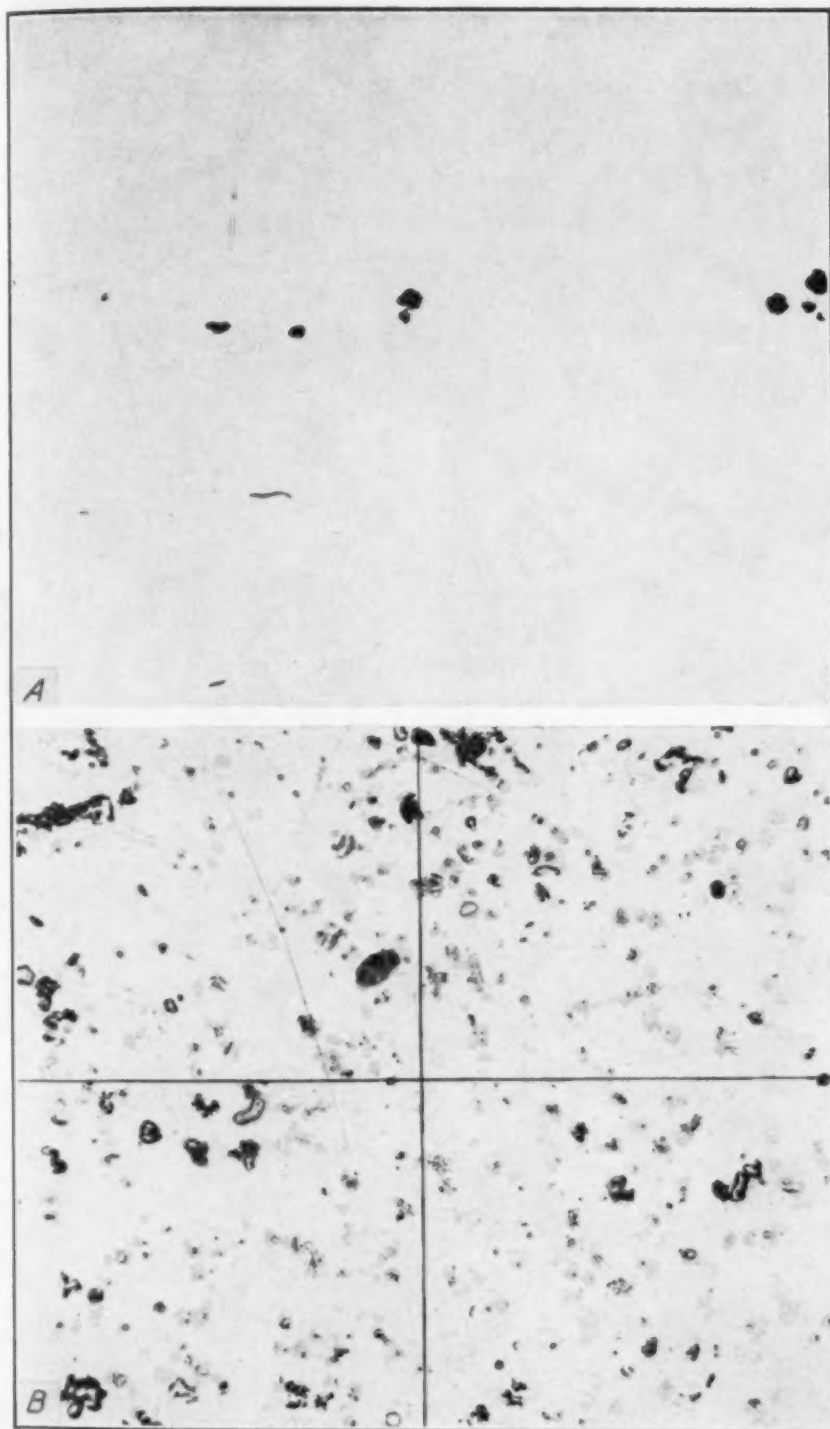


Fig. 4—Metallographic and Petrographic Photographs of Sample B.  $\times 200$ . (A) Metallographic Sample. (B) Petrographic Sample.

$\text{Al}_2\text{O}_3$  may be seen. Many fine irregular pieces of glass are also visible, together with several larger pieces of glass.

Fig. 5 is of interest because of the presence of the phase we

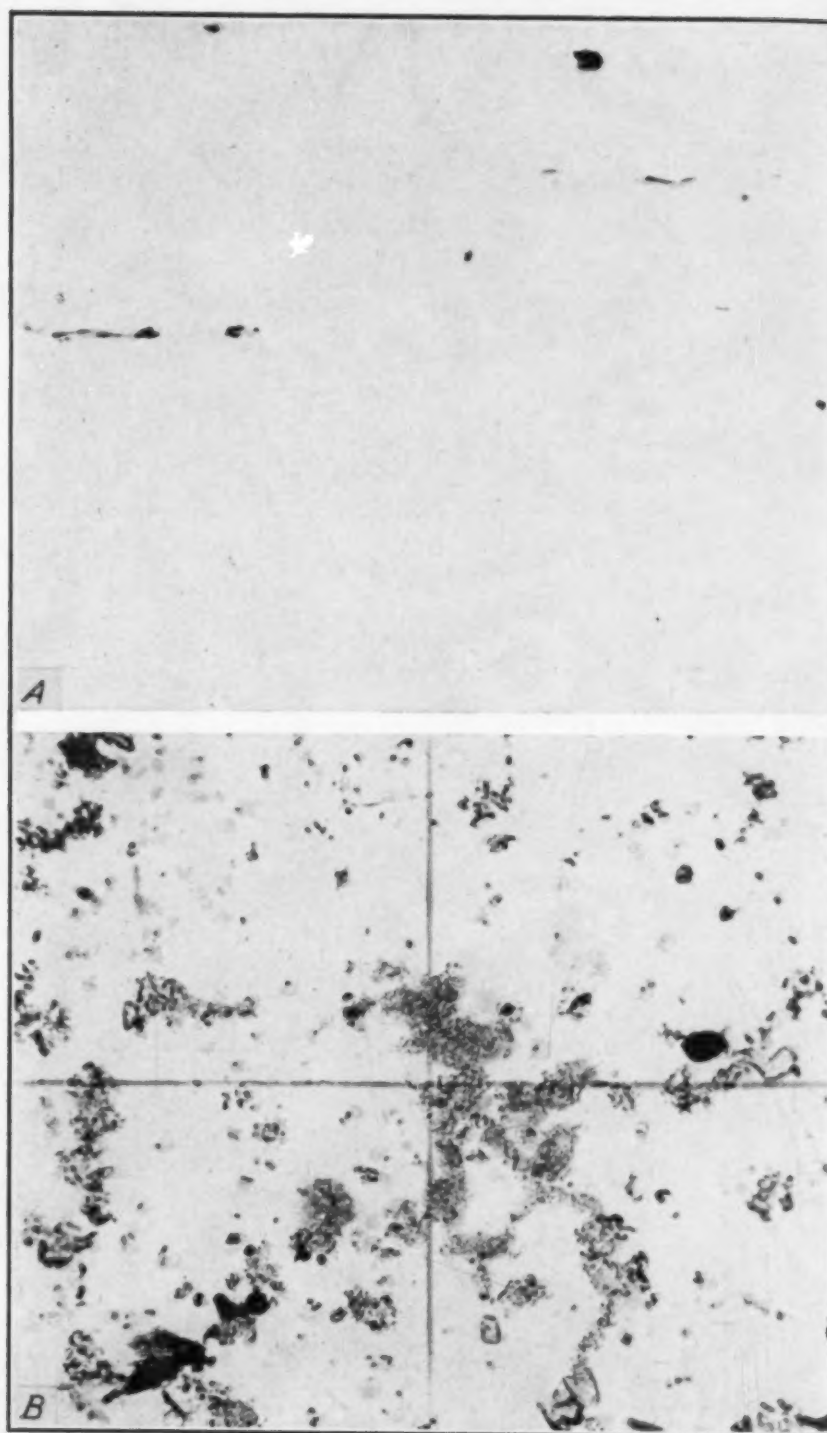


Fig. 5—Metallographic and Petrographic Photographs of Sample C.  $\times 200$ . (A) Metallographic Sample. (B) Petrographic Sample.

consider to be of fireclay origin. This phase is the extensive clumped material near the cross hairs in the petrographic photomicrograph. It is not shown in the metallographic section, where the inclusions are



composed of rounded  $\text{Al}_2\text{O}_3$  particles and numerous silicate stringers.

Fig. 6 shows a heat containing both  $\text{Al}_2\text{O}_3$  particles arranged in stringer form and silicate stringers. The petrographic photomicrograph shows  $\text{Al}_2\text{O}_3$  particles and pieces of glassy phase.

Fig. 7 shows a heat of fairly high  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  ratio. Both the silicates and  $\text{Al}_2\text{O}_3$  particles are present in stringers. These are well shown in the metallographic photomicrograph. Fragments of glass from the stringers and individual  $\text{Al}_2\text{O}_3$  inclusions are shown in the petrographic photomicrograph.

Figs. 8, 9 and 10 illustrate heats of fairly high  $\text{Al}_2\text{O}_3$  content, the inclusion being largely composed of the unique Phase 6. In Figs. 8 and 10 practically no stringer inclusions appear in the metallographic sections and likewise practically no glass particles are found in the petrographic photomicrographs. In Fig. 9, however, some of the  $\text{Al}_2\text{O}_3$  content appears as Phase 3 glass, seen as clumps in the petrographic photomicrograph, with short stringers also showing.

Petrographic study shows the Phase 6 in inclusions to be nearly pure aluminum oxide. However, instead of occurring in small individual particles, this phase is found in clumps or round masses made up of several individual corundum particles sintered or cemented together like a popcorn ball. The formation of these clumps instead of individual particles is a result of suitable furnace practices. The metallographic micrograph shown in Fig. 10a shows the complex nature of these inclusions as do the several petrographic photomicrographs. To illustrate their nature more clearly, Fig. 11 shows a photomicrograph of a petrographic sample of these inclusions at 635 diameters. These clumps of alumina particles may reach 0.001 inch in diameter in some cases.

It will be seen from the photomicrographs that in the cases where corundum clusters are obtained, very few aluminum oxide stringers are noted in spite of the generally higher  $\text{Al}_2\text{O}_3$  percentage. This may be attributed to the lack of minute  $\text{Al}_2\text{O}_3$  particles. Fig. 7, however, shows considerable evidence of aluminum oxide stringers as well as an absence of the Phase 6 agglomerate, indicating the importance of controlling the nature of the inclusions by suitable furnace practice.

X-ray powder diffraction photographs of the eight inclusion samples listed in Table I were made, using pure mullite and corundum as standards. The occurrence of these phases as noted by petrographic analysis was confirmed.

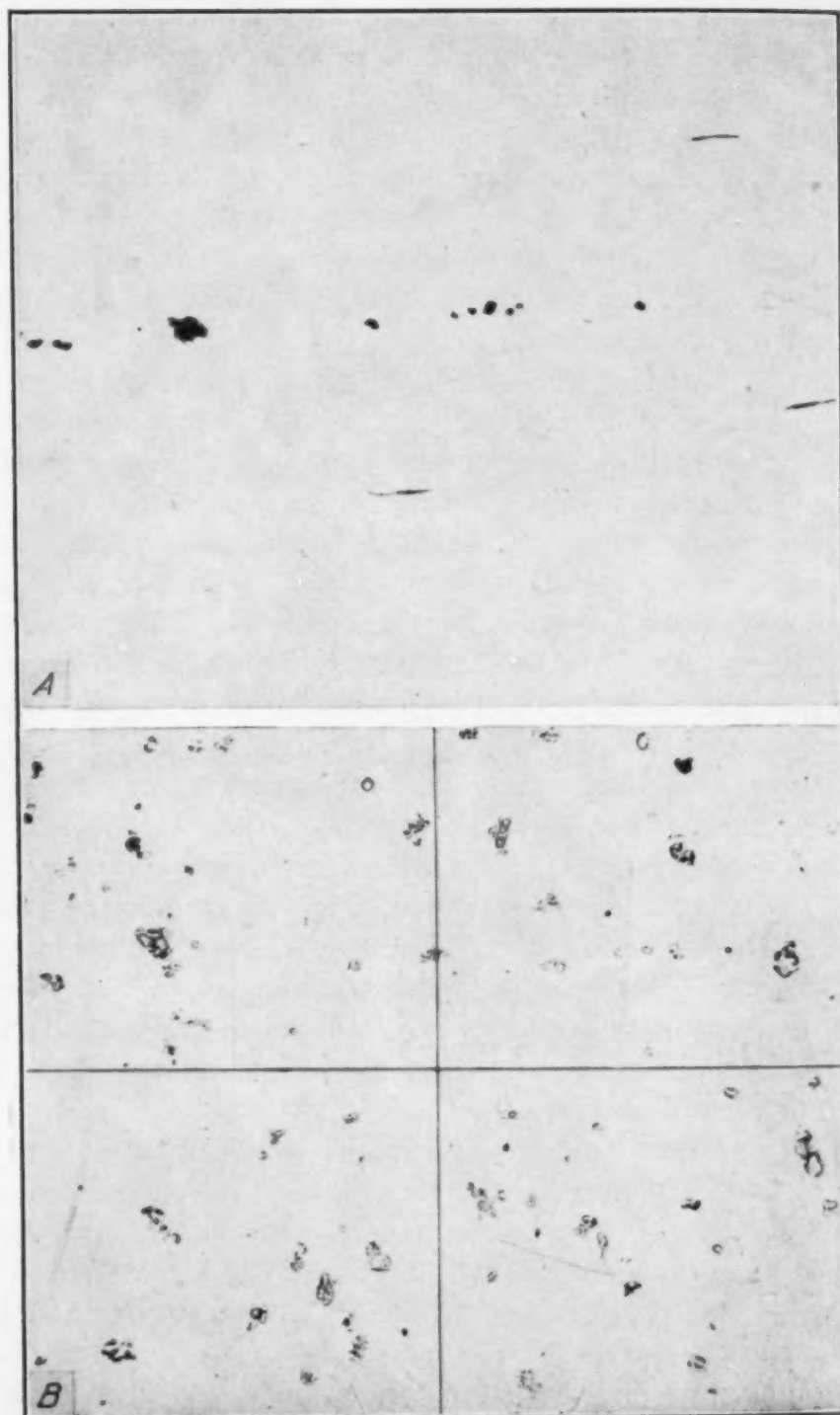


Fig. 6—Metallographic and Petrographic Photographs of Sample D.  
× 200. (A) Metallographic Sample. (B) Petrographic Sample.

#### FIRECLAY INCLUSIONS

Before considering the application of this work to specific types

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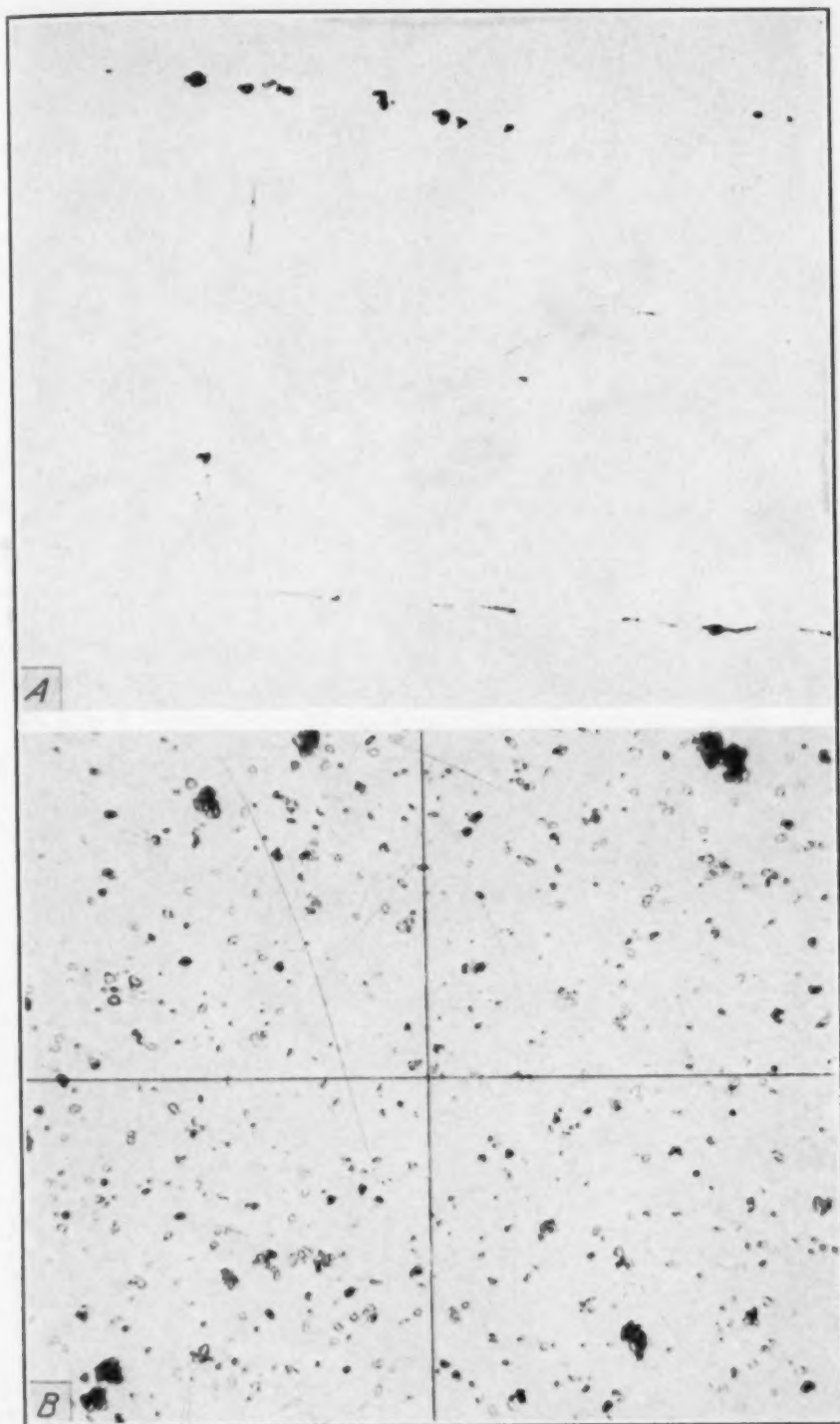


Fig. 7—Metallographic and Petrographic Photographs of Sample E.  
× 200. (A) Metallographic Sample. (B) Petrographic Sample.

of steel, another type of inclusion should be discussed. In the process of steel manufacture, particles of refractory material are occasionally carried into the molten metal. If they do not rise out by their

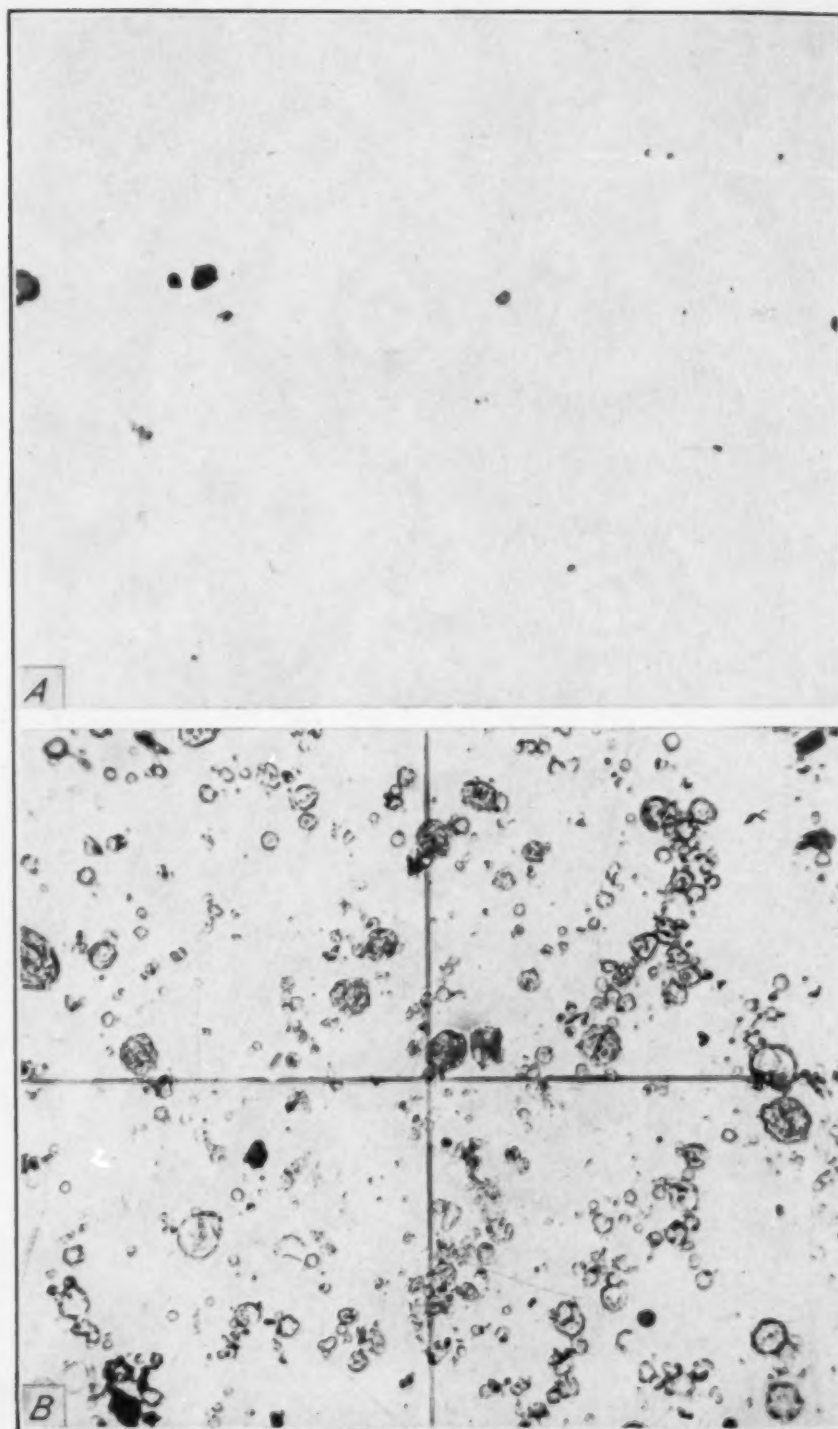


Fig. 8—Metallographic and Petrographic Photographs of Sample F.  $\times 200$ . (A) Metallographic Sample. (B) Petrographic Sample.

buoyancy they will be found in the finished product. These inclusions are generally much larger than those resulting from deoxidation products, and although of relatively infrequent occurrence,

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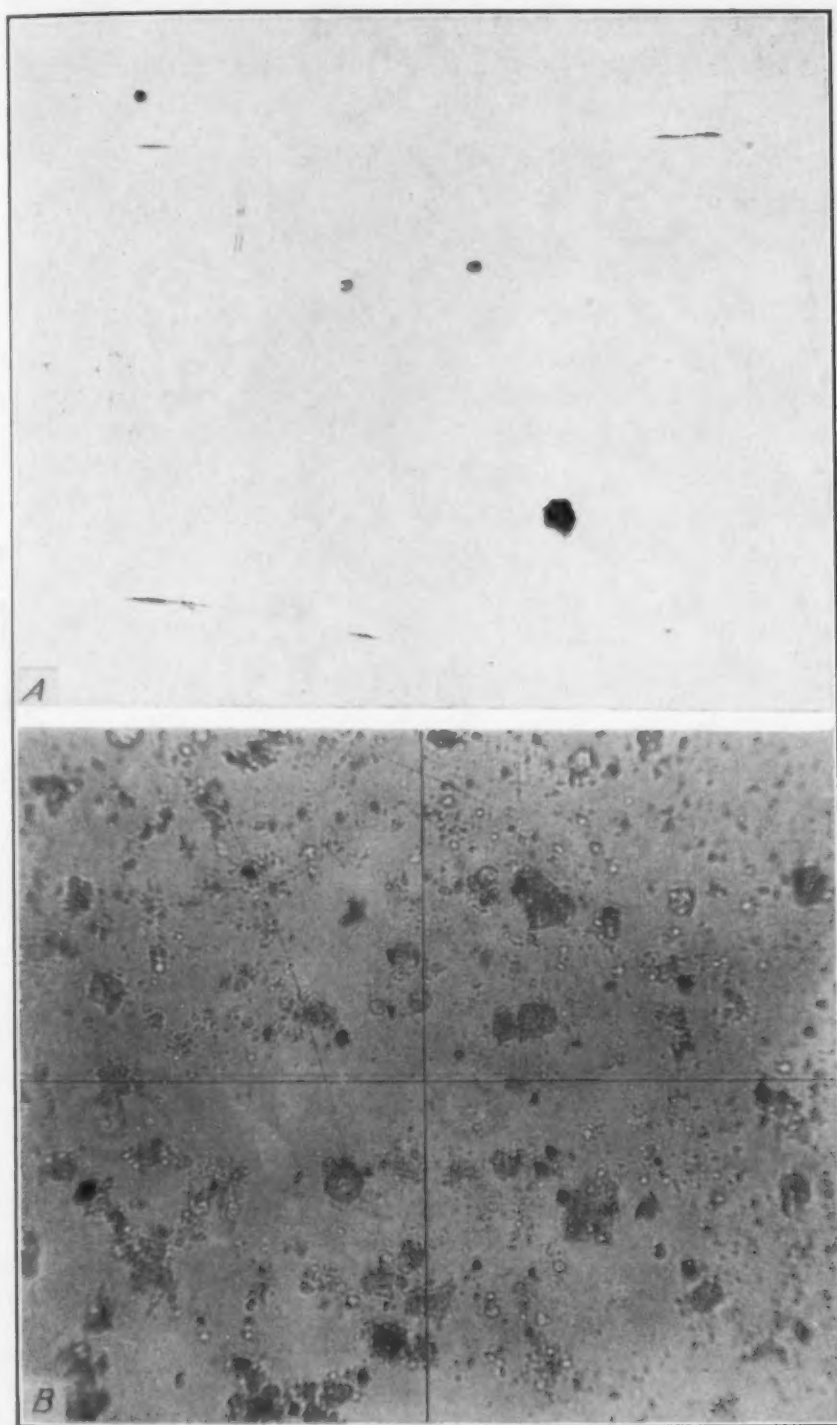


Fig. 9—Metallographic and Petrographic Photographs of Sample G.  $\times 200$ . (A) Metallographic Sample. (B) Petrographic Sample.

cause a certain amount of trouble during machining of the steel. Several such inclusions have been studied either by digging them out of the steel or by separating them electrolytically. Analysis of

these inclusions indicates them to be of fireclay origin, but frequently modified in composition by reaction with FeO or products of deoxidation. Petrographic study shows that they are characterized by the Phase 4, previously noted, together with such other phases as may be present due to inter-reaction with deoxidation products. One such inclusion, for example, showed a zone containing crystals of  $\text{FeO} \cdot \text{Al}_2\text{O}_3$  (Hercynite) in addition to the unaffected fireclay phase. Other inclusions of this type will contain needle-like crystals of mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ).

Fig. 12 shows a petrographic photomicrograph of a typical fireclay inclusion which is preponderantly composed of Phase 4. The clumped material shows the "fish-egg" appearance of this phase. The similarity of the material in this known fireclay inclusion and of the material noted in Fig. 5 is clearly evident.

#### RELATION OF INCLUSION PHASES AND EQUILIBRIUM DIAGRAM

It is interesting to consider the phases observed in this inclusion study in the light of the possible compounds and glass phases to be expected from the ternary equilibrium diagram  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot (\text{FeMn})\text{O}$ . H. Löfquist<sup>4</sup> has done extensive work on the inclusion phases found in polished sections of samples of steel and wrought iron, variously deoxidized. Snow<sup>5</sup> has also recently studied the system  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{FeO}$  with respect to the various compounds present and the index of refraction of certain glass compositions. The system  $\text{FeO} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  is shown after Snow in Fig. 13. MnO is not present in this system, but to a certain degree we may consider MnO and FeO as interchangeable without too great a change of mineralogical properties. In the system shown, the following compounds may be listed: Corundum ( $\text{Al}_2\text{O}_3$ ), Mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ), Cristobalite (tridymite, etc.) ( $\text{SiO}_2$ ), Hercynite ( $\text{FeO} \cdot \text{Al}_2\text{O}_3$ ), and Fayalite ( $2\text{FeO} \cdot \text{SiO}_2$ ). In the presence of much MnO the metasilicate phase  $(\text{FeMn})\text{O} \cdot \text{SiO}_2$  is also possible.

In our studies of deoxidized steels, we have observed Corundum and Mullite but have not noted either Cristobalite or Tridymite. The quartz found as inclusions has not crystallized from melts but must have found its way into the steel from fireclay refractories or other sources. Hercynite has been observed as remarked earlier and the metasilicate  $(\text{FeMn})\text{O} \cdot \text{SiO}_2$  has been tentatively identified but not

<sup>4</sup>H. Löfquist, *Jernkontorets Annaler*, Vol. 117, 1933, p. 49-115.

<sup>5</sup>R. B. Snow, Doctor's Dissertation, Ohio State University, 1936.

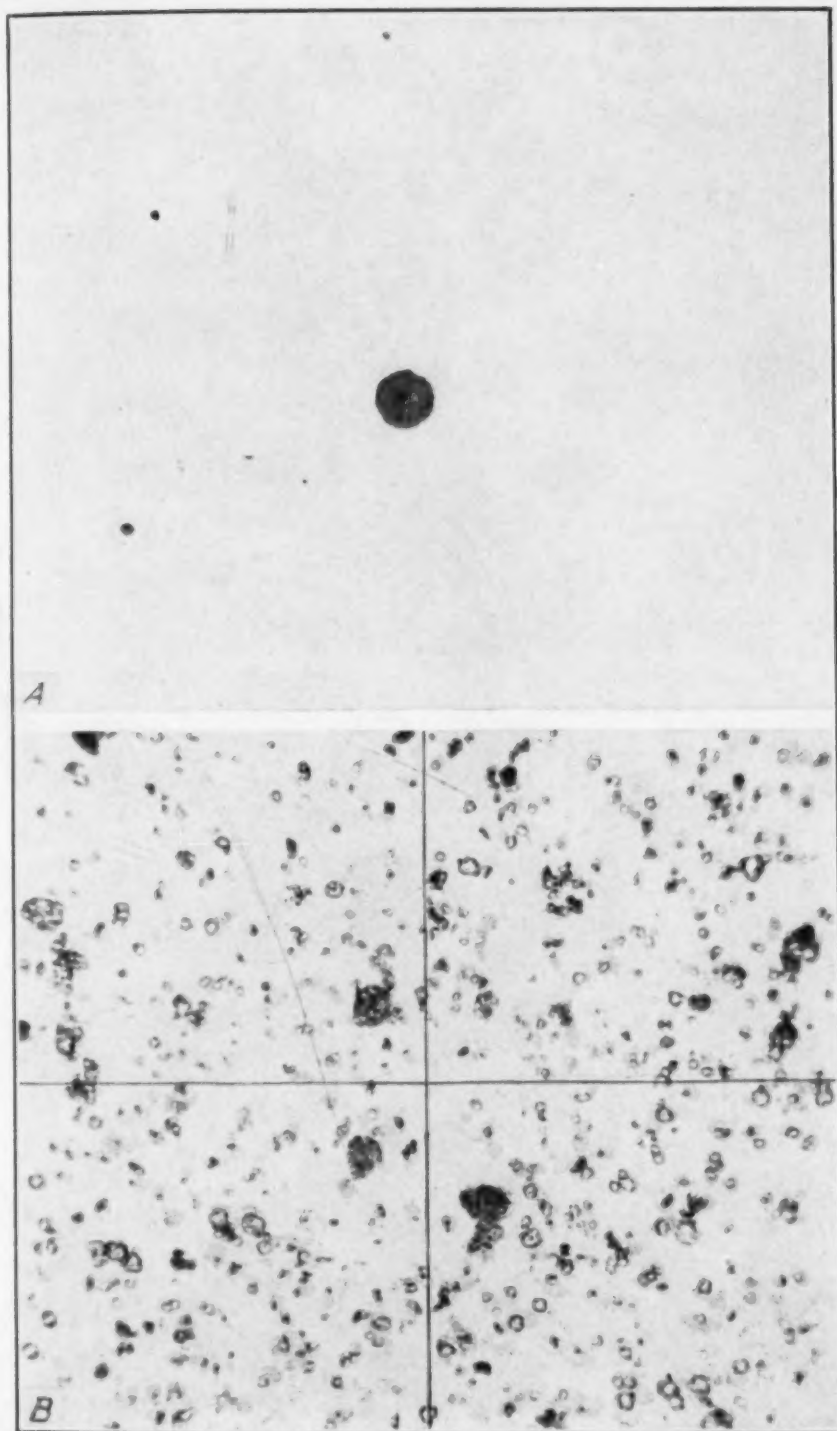


Fig. 10—Metallographic and Petrographic Photographs of Sample H.  $\times 200$ . (A) Metallographic Sample. (B) Petrographic Sample.

frequently observed. The crystal phases of higher FeO, MnO content have not been observed.

Snow has also studied the index of refraction of some of the



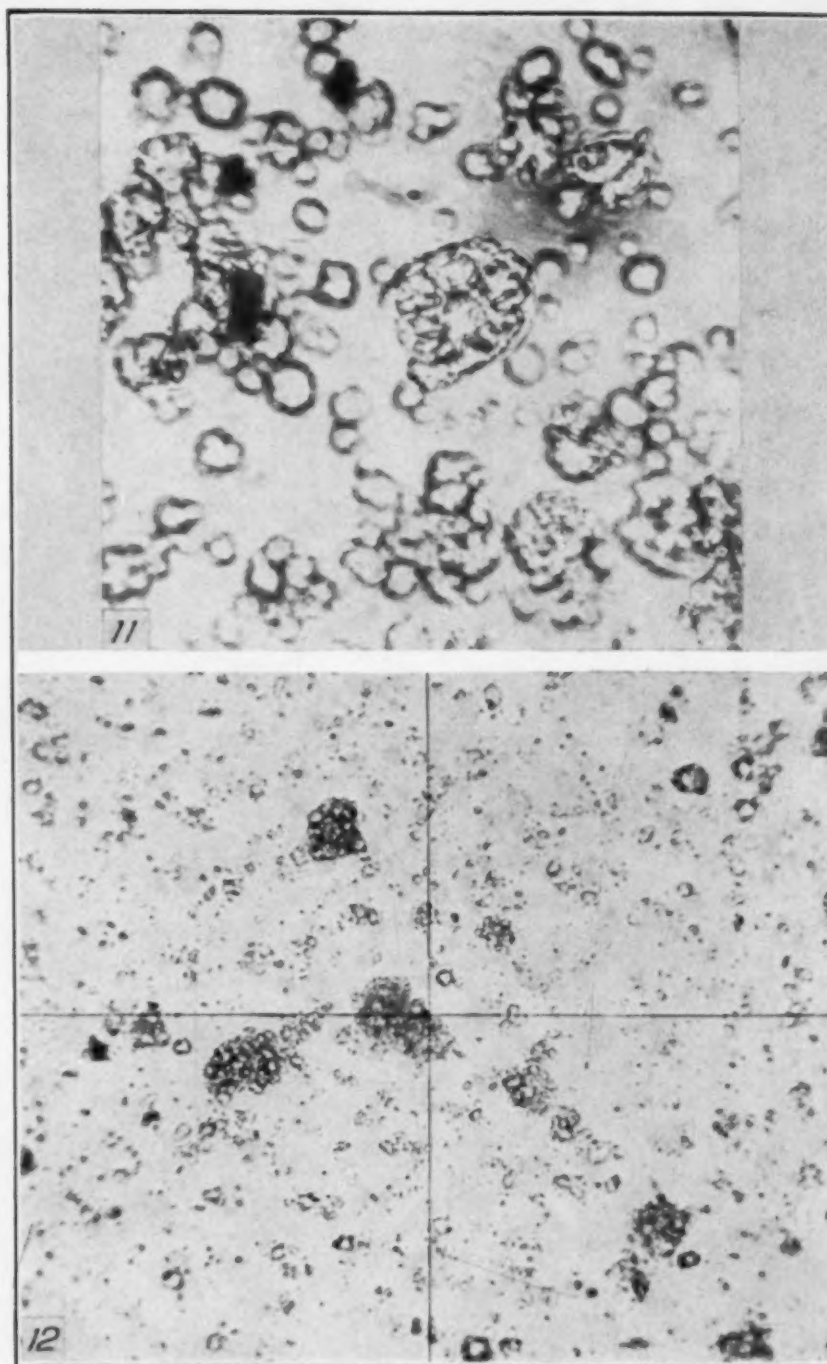


Fig. 11—Phase No. 6 Inclusions.  $\times 635$ . Showing Structure of  $\text{Al}_2\text{O}_3$  Agglomerates.  
Fig. 12—Petrographic Micrographs of Separated Fireclay Inclusions.  $\times 200$ .

glass compositions in the system  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{FeO}$ , and additional data for the binary system  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  have been obtained by Lu.<sup>6</sup> A diagram of this system including these data is given in Fig. 14,

<sup>6</sup>Kai Ching Lu, Doctor's Dissertation, Ohio State University, 1928.

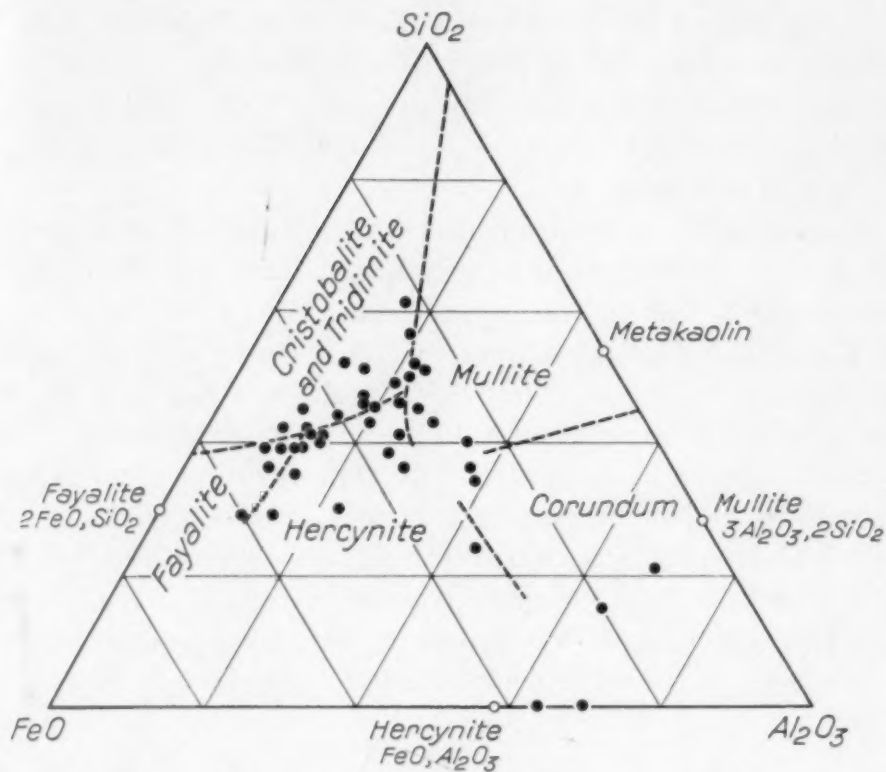


Fig. 13—Ternary Diagram of FeO:Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub> (Snow<sup>5</sup>).

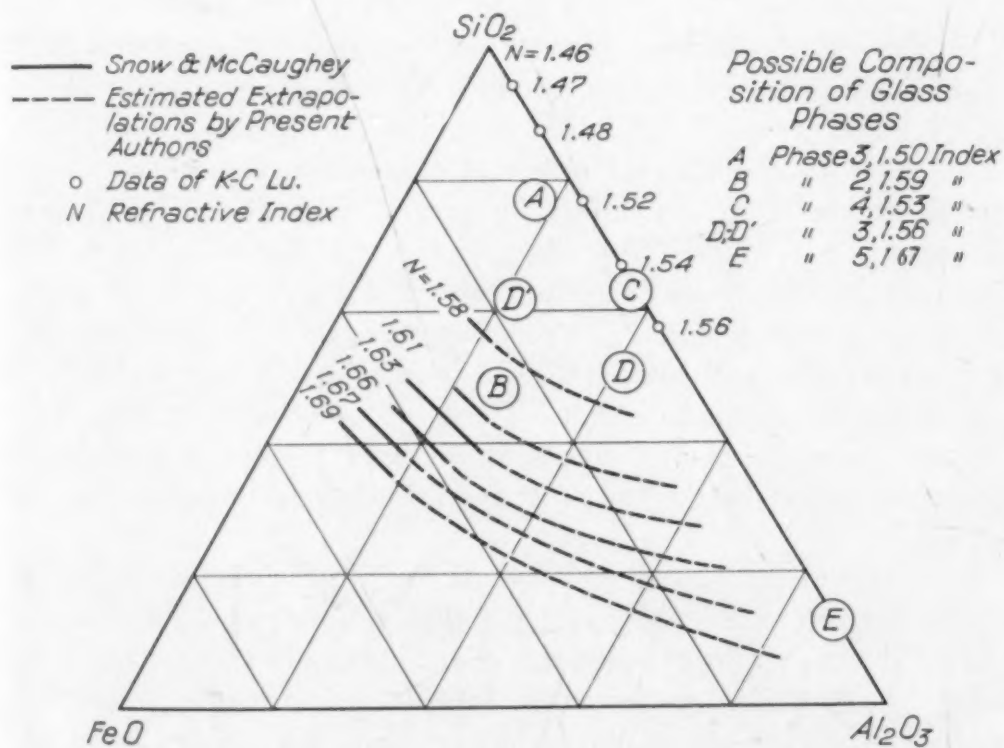


Fig. 14—Ternary Diagram of Al<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub>:FeO. Index of Refraction of Glass Phases.

with extrapolations of our own to allow estimates of the glass refractive index over a wider range of composition. Since FeO and MnO have similar properties and affect the glass indices similarly, we have assumed that we may use these two oxides as a single component in this diagram.

In thoroughly deoxidized steels, it is probable that the glass phases are largely composed of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  with less FeO and MnO. Samples less thoroughly deoxidized may contain glasses of much higher (FeMn)O content. The index of refraction alone determines only a line of composition and not a point so that there will be some error in attempting to place the composition accurately. However, taking into account the over-all chemical composition of the inclusions and other pertinent factors, we have made estimates of probable compositions of several glasses observed as inclusions. These are indicated on the graph shown in Fig. 14. An inspection of the data will indicate the degree of accuracy of these estimates and the range of possible composition of the several glass phases observed.

#### EFFECT OF FURNACE PRACTICE ON INCLUSIONS

The effect of varied practice on the type and amount of inclusions formed in steel is illustrated in Table II. The electric furnace heats listed are of S.A.E. 4615 specification. Heat AA represents steel made by a somewhat elaborate experimental practice in which the steel was carefully killed by several additions of silicon and aluminum. It will be observed that the total inclusions were quite low and that they were preponderantly  $\text{Al}_2\text{O}_3$  particles with a smaller amount of glass phase. The next set of heats BB-FF represents a more simple deoxidation practice in which the silicon is added in the furnace with aluminum at tap. These heats show rather variable inclusion contents, due to variations in other factors, but in general contain a somewhat higher amount of inclusions than the first heat shown.

In one case, CC, for some reason the  $\text{SiO}_2$  was abnormally high. The reason for this was possibly that the heat was colder than the others shown, with the result that the silicates did not rise out of the steel. In any case, the increased  $\text{SiO}_2$  is shown in the petrographic ratings by the large percentage of glassy material. In the last column the analyzed values of per cent FeO present in the several heats





before aluminum additions are shown and it will be noted that they are fairly low.

The third set of heats at the bottom of the table represent a practice in which silicon is added in the ladle with little deoxidation prior to the aluminum addition made before tapping. Several things will be noted in this set. The per cent FeO in the steel before aluminum addition (last column) is relatively high, as might be expected. Consequently, the per cent  $\text{Al}_2\text{O}_3$  and hence the total amount of inclusions are somewhat higher than in other cases. Petrographically, the inclusions from these heats are composed largely of corundum, but it will be noted that this material appears to a considerable extent in the agglomerate or Phase 6 form rather than as individual particles. These heats were therefore largely free from stringer inclusions.

The very fair correlation between the per cent dissolved FeO

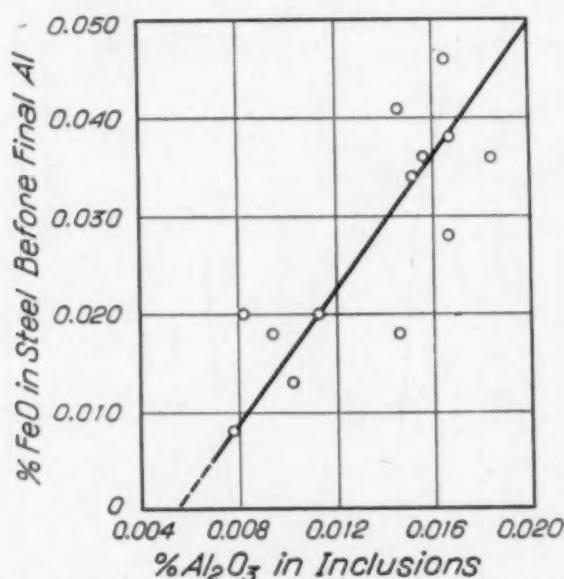


Fig. 15—Effect of Degree of Oxidation on Alumina Inclusions.

in the steel at tap as determined by the gas analysis method and the per cent  $\text{Al}_2\text{O}_3$  inclusions formed may be noted in Table II and in the graph shown in Fig. 15. The spread of points is due to the oxidation of residual aluminum during pouring, etc., but the trend is clear. However, the extrapolation to the horizontal axis probably roughly indicates the amount of  $\text{Al}_2\text{O}_3$  formed by the action of air on residual aluminum during tapping and pouring, being around 0.005 per cent plus the amount of alumina which is formed and rises out of the bath.

## ACKNOWLEDGMENTS

The authors wish to acknowledge the assistance of C. A. Stroup in the chemical separation and analysis of the inclusions and of R. K. Lee and J. E. Harris for the petrographic analyses and preparation of micrographs.

## DISCUSSION

**Written Discussion:** By Frank G. Norris, open-hearth metallurgist, Wheeling Steel Corp., Steubenville, O.

When I read the paper by Hare, Peterson and Soler in a recent issue of *TRANSACTIONS*, I regretted that no opportunity for discussion of this paper had been given. This is an interesting paper and will doubtless pave the way for the accumulation of data on evolved gases concerning which there has been much speculation but few facts. Since the authors have alluded briefly to their former paper, a brief discussion of it will not be out of place at this time.

The method of taking duplicate samples from the bath offers a good opportunity to check the sampling procedure as well as the chemical determination. The authors are to be congratulated on the agreement shown in Table I which is quite satisfactory. The agreement is indeed closer than is often possible when the duplicate determinations of FeO are both made by the same method. The results of both methods are in the general range that is found in a bath containing 0.07 to 0.10 per cent carbon. Does this indicate that the  $\text{FeO} \times \text{C}$  product is about 10 times as great for high carbon as for low carbon heats? For low carbon heats the product is about 0.014. The product of the values in Table I is in the range from 0.112 to 0.188.

Considering the  $\text{FeO} \times \text{C}$  reaction as a basis for the determination of FeO, it should make no difference whether the CO evolved or the C oxidized is determined. For the purpose of ordinary production control the determination of carbon on killed and unkilld samples seems simpler than the determination of the CO evolved.

Assume that the carbon determinations are made on a killed sample with the last sample containing 0.685 per cent carbon, and 0.178 per cent FeO. This amount of FeO is equivalent to 0.030 per cent carbon and the live sample would be expected to contain 0.655 per cent carbon. If the 0.685 per cent carbon was determined on a live sample, of course the correction would be added to give 0.715 for the killed sample. This method of using the difference in carbon as a measure of FeO is, of course, subject to greater error at high carbon values because carbon determinations may not be sufficiently accurate. The FeO is of greatest interest, however, near the end of the heat when the carbon is lower.

Referring to the relation between FeO before the aluminum addition and the type of inclusions formed, is the value of the FeO content of chief importance or is the manner of arriving at this value of some effect? The FeO content of the bath can be modified by the nature of the slag, the preliminary deoxidation or combination of the two. It is possible that in a certain range the same FeO could result from each of these 3 methods. In this case, could the cheapest and most convenient method be used with the assurance that the effect



of the FeO content is independent of the practice used to bring the bath to this level?

**Written Discussion:** By Frank W. Scott, Instructor of Ferrous Metallurgy, University of Minnesota, Minneapolis.

In 1935, when Mr. Hare visited with me at Inland Steel Co. to compare notes in regard to the electrolytic method of oxide extraction and analysis, we were in agreement that oxide determinations had more importance than merely the determination of the oxygen present in steel. While the vacuum fusion method, or hydrogen reduction method, would furnish data regarding the total oxygen present, it was considered more necessary to know in what manner this oxygen occurred, and so determine the source of the oxidic particles. Knowing this source, some steps could be taken to reduce the amounts present, or to produce the types desired; as for instance, those in certain grain size controlled steels. In this paper, Messrs. Hare and Soler show how their company is striving to improve quality by using all the scientific means available in this study, by (1) extraction of oxide inclusions without destructive attack by the iron solvent or contamination by metalloids compounds; (2), petrographic and X-ray identification of these inclusions; (3), distribution in steel by use of the metallographic microscope. With first-hand knowledge of the care and precision necessary to carry out such a program, the results of which are here reported, I wish to congratulate the authors on the completion of the work and its able presentation.

Being well acquainted with this method of oxide separation, there are two points in this revised method that seem open to criticism, or suggestion. In the first part of the procedure, after the oxides are extracted, Mr. Hare states that the residue, carbon, oxides, sulphur, and perhaps some silicic acid, is washed into a beaker with water, "heated gently," and filtered, being washed with hot water. It is known that ferrous iodide oxidizes readily, and that it decomposes in hot water to form iron hydroxide, ferrous hydroxide at first and then ferric hydroxide. In such a form the subsequent treatment will not remove the hydrate from the residue, and it will be assumed to be part of the oxide inclusions, or it may be thought to come into the residue during the electrolysis, which is not the case. So I would suggest the following procedure.

Referring to the results in Table I, the highest FeO analysis is seen to be in the highest carbon ranges. This might be construed to mean that the carbides are not decomposed, but work that I have done on pig iron and cast iron has shown that carbides are completely decomposed. Rather it means that the carbon has absorbed large amounts of iron salts that can be removed only by leaching in citrate or tartrate solutions, and no opportunity must be afforded for this ferrous iodide to decompose. Direct washing is not sufficient and the residues must be allowed to leach over night, or longer, with washing. The first washing should be done with citrate or tartrate solutions, and if the residue is heated, it should be in this solution. If this procedure is adopted, I feel sure that the FeO content of the inclusions will no doubt conform to their petrographic classification; as for instance, referring to Table I, sample E, the FeO probably should be of the same order as the MnO, or about 0.003 per cent.

My second comment is upon the use of hydrogen in the extraction cell to



prevent the action of air on the iron salts. During the electrolysis, hydrogen is given off at the cathode where some magnesium hydroxide is formed. With a close fitting top on the cell there is nearly always enough gas formed to prevent access of air, and so oxygen. My experience has shown such precautions as Mr. Hare has adopted are not required, though, of course, they do no harm.

A general comment might be made in regard to the problem of deoxidation type inclusions in carbon steels and their relation to furnace practice. About three years ago we made several hundred oxide determinations at Inland Steel Co. on tapping tests at the furnace, tests taken at the pouring stand, and later on billet tests. These were correlated to slag conditions, deoxidation practice, and to microscopic inclusion counts. As a result, we established what might be termed the expected cleanliness for any deoxidation practice, and so could classify heats according to the average of these tests. Steps were taken to change the practice where steels were abnormally contaminated with these oxides. The effect of high and low iron oxide slags were evaluated. The effect of pig iron cleanliness upon the steel was determined. While for obvious reasons these results were not published, the practical application of this method of oxide determination was then demonstrated. The authors have demonstrated its application to certain alloy steels. It is my hope that other research metallurgists may find this method of inclusion study equally satisfactory, and that it may generally be adopted for such determinations.

#### Authors' Reply

Mr. Norris' discussion refers mainly, as he states, to the method of analysis of the gases in molten steel, reported recently from our laboratory and referred to in the present paper.

While we originally hoped to present this method and discuss the results at the convention, we did not for several reasons get the article ready in time for presentation. Additional results of the method will, however, be published in the *TRANSACTIONS* shortly.

It is true that the values we obtain for per cent CO from steels of medium and higher carbon content indicate that FeO and carbon are present in these steels considerably in excess of the equilibrium value usually given in the literature for low carbon steels. We consistently obtain products of  $(\text{FeO}) \cdot (\text{C})$  of around 0.100 to 0.150 or more during the working or oxidized portion of open-hearth and electric heats as long as the carbon is over 0.60 per cent or so. We hope to study this reaction in more detail at some time, but are of the opinion that the results are fairly accurate. There are at least two reasons why these results should not appear very surprising. In the first place, these tests which show a high product of  $(\text{FeO}) \cdot (\text{C})$  at high carbon contents are obviously not at equilibrium since the carbon is always dropping, usually fairly rapidly. As Schenck has indicated (*Stahl und Eisen*, Vol. 41, 1049, 1933), the product  $(\text{FeO}) \cdot (\text{C})$ , when in excess of the equilibrium value measures the rate of carbon drop. Possibly the actual product of  $(\text{FeO}) \cdot (\text{C})$  for 1 per cent carbon would at equilibrium be nearer the value shown in Table II of the article referred to, for undeoxidized steel under a carbide slag, namely about 0.040 per cent or so.

In the second place it has been previously stated that the actual equilibrium produce  $(\text{FeO}) \cdot (\text{C})$  is higher for higher than for lower carbon contents, due probably to a lowered activity of the carbon at high concentrations. Herty on page 29 states this fact in "The Control of Iron Oxide in the Basic Open Hearth Process."

Norris' suggestion that the carbon content on killed and open samples be run as a measure of per cent FeO is interesting. This method would be quite rough, however, since the difference between the two carbon contents would only be about 0.03 per cent carbon, as he states, and an error of even 0.005 per cent carbon would make a percentage error of 16 per cent. Moreover, there would be the question as to whether the same amount of CO is evolved from an open sample frozen at atmospheric pressure as from a sample frozen in an evacuated tube.

In regard to Mr. Norris' question as to the relative importance of the per cent FeO at final deoxidation and the manner of arriving at this value, we would say that the per cent FeO before Al addition is the most important factor, since this will determine the per cent  $\text{Al}_2\text{O}_3$  formed. However, the presence of silicates suspended in the bath due to prior silicon deoxidation might readily affect the nature of the aluminum formation by forming, say,  $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$  glasses. For controlling the more subtle physical properties of steel such as hardenability, etc., it is probable that one would find that not only the per cent FeO at final deoxidation but also to a less extent the manner of arriving at this per cent FeO would be of importance.

We wish to thank Mr. Scott for his remarks and suggestions. We are in entire agreement with his estimate of the utility of the method for establishing practices and checking the effect of varied furnace practice on oxide content and properties of steel.

Mr. Scott has called attention to a point of technique in regard to washing the residue after electrolysis, stating that the first washing should be with a tartrate solution to prevent oxidation of iron salts.

We are glad he called our attention to this. We actually employed this precaution but due to an unfortunate oversight omitted it from the description of the procedure in a previous article. The procedure was to add sodium tartrate solution to the residue immediately after washing it into the beaker, then after warming it, to filter and again treat the residue overnight with tartrate solution.

It is our opinion, however, as stated on the sixth page of the paper, that the per cent FeO are still somewhat high due to impossibility of absolutely removing all iron salts. The results we obtained on the Bureau of Standards samples seem to show, however, that the technique employed gave as low values of per cent FeO as those obtained by any analyst who employed a comparable method.

We have used nitrogen gas during electrolysis as a precaution in running these tests since we would otherwise occasionally note some oxidation of the surface of the electrolyte. We have employed a somewhat lower voltage, I believe, than Mr. Scott, and in general would not, therefore, obtain sufficient hydrogen evolution to prevent possible oxidation.

## SOME PROBLEMS IN THE PRODUCTION OF LOW CARBON SHEETS IN NON-CONTINUOUS MILLS

By M. L. SAMUELS AND ALFRED BOYLES

### *Abstract*

*The failure of sheets in cold forming is discussed from the standpoint of grain size. Examples are shown of failures due to very fine grain as well as to coarse grain. Among the factors influencing the degree of strain produced by rolling, two are pointed out in particular:*

- 1. The effect of segregation in the core of rimmed ingots.*
- 2. The effect of the grain size of the sheet bar previous to rolling into sheets.*

*The view is advanced that phosphorus, by increasing the stiffness of the metal at finishing mill temperatures, increases the degree of strain and consequently produces a finer grain size after box annealing. Fine-grained cores in sheets from the upper part of the ingot are attributed in part to this cause.*

*Experiments are described illustrating the effect of the grain size of the sheet bar before rolling on the grain size of the sheet after box annealing. It is thought that a coarse-grained sheet bar is strained less in rolling than one having a fine grain. Irregularities in structure in the finished sheet may sometimes be traced to exaggerated grain growth on the surface of sheet bars due to their having been stacked hot at the bar mill.*

THE sheet steel industry is undergoing an evolution which started with the installation of two 48-inch continuous strip mills in 1926 by the American Rolling Mill Company. The growth of the continuous mill capacity during the last decade has been phenomenal and now the annual capacities of continuous sheet and wide strip mills installed and under construction in the United States has reached approximately 12,000,000 tons.<sup>1</sup> It has been freely predicted that the installation of continuous mills spelled the doom of non-continuous hand mills, but it must be admitted that these mills

<sup>1</sup>*Iron Age*, January 7, 1937, p. 134.

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. The authors are metallurgists, Battelle Memorial Institute, Columbus, Ohio. Manuscript received April 13, 1937.



are still running close to capacity. The ability of the non-continuous mill to change quickly from one order to another, to fill economically small orders of only a few hundred sheets, and to roll special sizes and grades will enable this type of mill to hold a place in the industry, provided the management keeps production costs and quality of product properly balanced.

There are many metallurgical problems in common between the non-continuous mills and the continuous strip mills, but, also, there are some important points of difference. The writers wish to discuss in this paper some of the problems with particular reference to the non-continuous mills and it is hoped that the present paper may throw at least a little light on some of the production factors which control cold-forming properties. The quality factor is of special significance in view of the competition, not only from similar producers but from continuous mills, and it is likely that the non-continuous mill operators who do not closely adhere to those principles which yield a high quality sheet will not long be in the field.

#### TYPES OF FAILURES

The sheet producer who is trying to improve the quality of his product to meet increasingly severe demands by the customer would do well to divide his difficulties into two groups and study one at a time. The first source of failures may be laid to the steel-producing division and the second may be laid to the routine processing operations in the sheet mill. Under the two general divisions, the sheet producer could classify all his difficulties—those rejections from piping, open surfaces, excessive hardness, enameling and galvanizing difficulties, and a host of other troubles which are met with constantly. However, it is our purpose in this paper to discuss only some of the factors contributing to failure during cold-forming. In those instances where the sheet is definitely at fault, two general types of failures can be recognized. First, there is the "rotten break" or granulated fracture of the type shown in Fig. 1. Secondly, we have the fine break which is usually associated with marked directional properties and which is illustrated in Fig. 2.

The failure shown in Fig. 1 is clearly the result of improper sheet mill processing and that shown in Fig. 2 is the result either of poor open-hearth practice or improper shear discard. Occasionally, fine breaks occur from underannealing, but, if the ordinary production routine is followed, these two general classifications will hold.



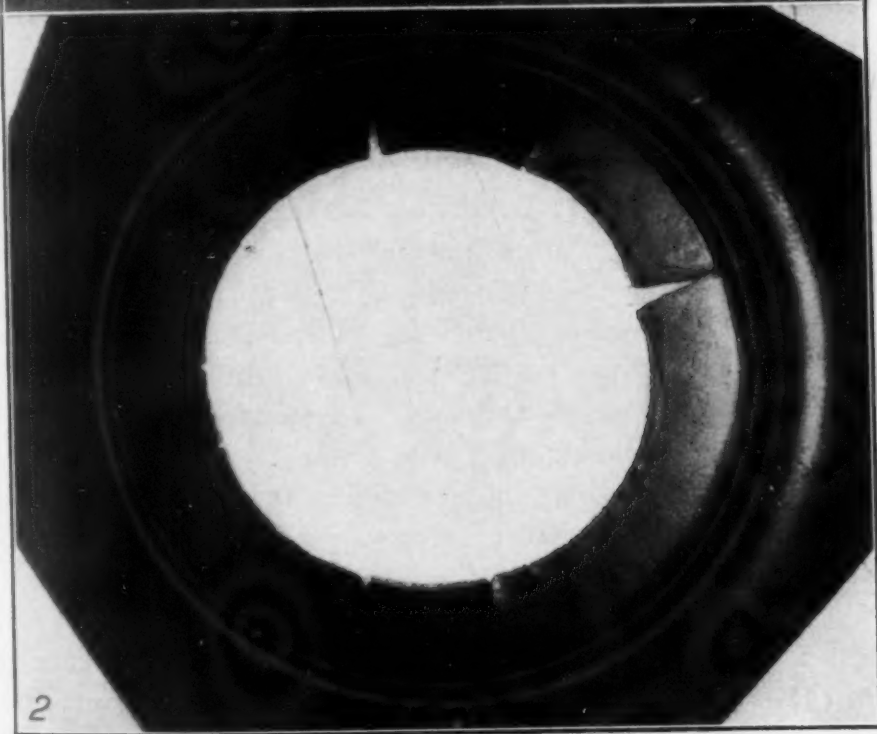
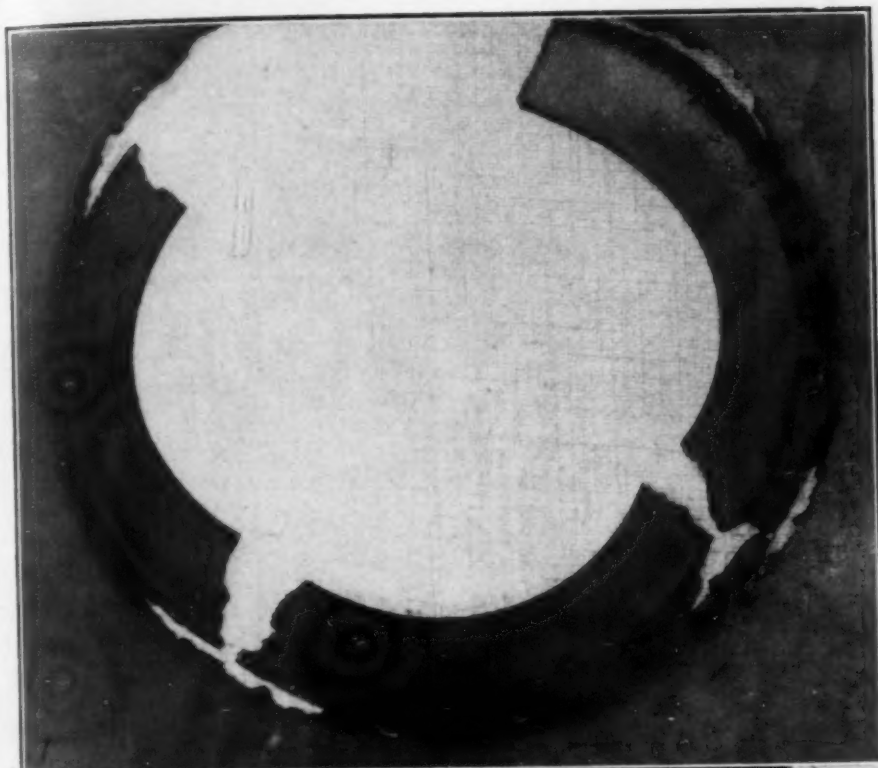


Fig. 1—Failure in Forming Bung Hole of Oil Drum Head.

Fig. 2—Another Failure in Forming Bung Hole of Oil Drum Head.

The failures shown in Figs. 1 and 2 were taken from oil drum heads which were made from 18-gage sheet of basic open-hearth steel. Additional data are given in Table I.

Table I  
Chemical and Physical Tests on the Two Types of Failures

Specimen	Analysis of the Sheet Near Failure Per Cent		Ladle Analysis Per Cent		Yield Strength Lb. Sq. In.	Ultimate Strength Lb. Sq. In.	Elong. in 2" Per Cent
	C	P	C	P			
Coarse Break—Fig. 1	0.04	0.015	0.08	0.019	17,200	33,000	26
Fine Break—Fig. 2	0.09	0.076	0.07	0.033	43,390	58,500	31

Photomicrographs (Figs. 5 and 6) show the structure of the fine-break material. When the analysis, physical properties and structures of the sheet are considered, it is easy to see why the failure of this particular piece occurred, but the conditions which gave rise to this type of sheet are not simple and they require a closer study.

#### SOURCE OF FINE-GRAINED FAILURES

The fundamental reactions in the open-hearth furnace are, of course, those of oxidation. It is by oxidation that carbon, phosphorus, silicon, and other impurities are removed from the liquid metal. In the making of rimmed steel the above-mentioned elements, which act as deoxidizers, are reduced to comparatively low values and there is a corresponding increase in the amount of iron oxide present. It is mainly the reaction of this iron oxide with the carbon in the steel that produces the strong gas evolution during the rimming period. The amount of the gas evolved during solidification plays a very important part in determining the number and location of blow holes and the degree of segregation within the ingot.

For purpose of discussion three types of ingots might be mentioned. In the first, the iron oxide content is low and the pressure of the gas formed is just sufficient to overcome the ferrostatic pressure in the mold, but the rate of evolution is not vigorous enough to sweep the gas bubbles away from the solid-liquid interface. As a result, numerous honeycomb blow holes form near the surface of the ingot and the metal rises in the mold, sometimes to a very marked extent. An ingot of this type is practically worthless for sheet bar

because the skin blow holes are opened up in the soaking pits, producing a very bad surface. The important consideration here is that, as far as segregation alone is concerned, this type of ingot is the best of all the rimmed group.

Upon going to a more highly oxidized and lower carbon steel a different type of structure is obtained. The skin over the honeycomb blow holes is thicker than in the first case, owing to a more rapid evolution of gas which prevents the bubbles from lodging until sufficient thickness has developed to slow down the rate of freezing and consequently of gas evolution. No honeycomb blow holes are found near the top of the ingot because the ferrostatic pressure imposes less restraint on the evolution of gas, and the bubbles, joined by those originating further down the ingot, are able to escape. This type of ingot rises less rapidly because of the larger quantity of gas escaping. The location of the primary blowholes in the lower portion of the ingot produces the effect known as the "high water" mark, due to the insulating action which prevents the rapid conduction of heat from the interior of the ingot. Shortly after stripping, the lower portion of the ingot is quite dark while the upper portion is still a bright red. This type is quite satisfactory for sheet bar because the skin is so thick that the primary blow holes are not opened up in the soaking pit. Major segregation, however, is noticeably greater than in the first type.

By increasing the amount of oxidation and lowering the carbon still more, a third type of ingot is produced (that characteristic of "low metalloid" heats). In this case gas evolution is violent and the metal drops in the mold. Almost no primary blow holes are formed because of the rapidity of gas evolution. A small pipe is usually found in the top which must be taken care of by a rather generous shear discard. Considerable quantities of aluminum have to be added to prevent foaming. Seams are almost entirely eliminated, but major segregation in this type of ingot is extreme.

The above three ingot types have been described to emphasize the fact that the production of sheets from rimmed steels is more or less a bargaining proposition. If one could consider segregation alone with utter disregard for seams and blisters, undoubtedly the first ingot type would be adopted. In order to improve the surface quality of the sheets, more segregation must be accepted and counter-balanced by keeping the interfering elements low and by proper shear discard.



Referring again to the fine break failure shown in Fig. 2, the logical explanation is that the phosphorus was too high. The ladle analysis was on the high side even for material intended to be used in a moderately difficult drawing operation and the heat should have been diverted to a lower grade product. Fine break failures, how-

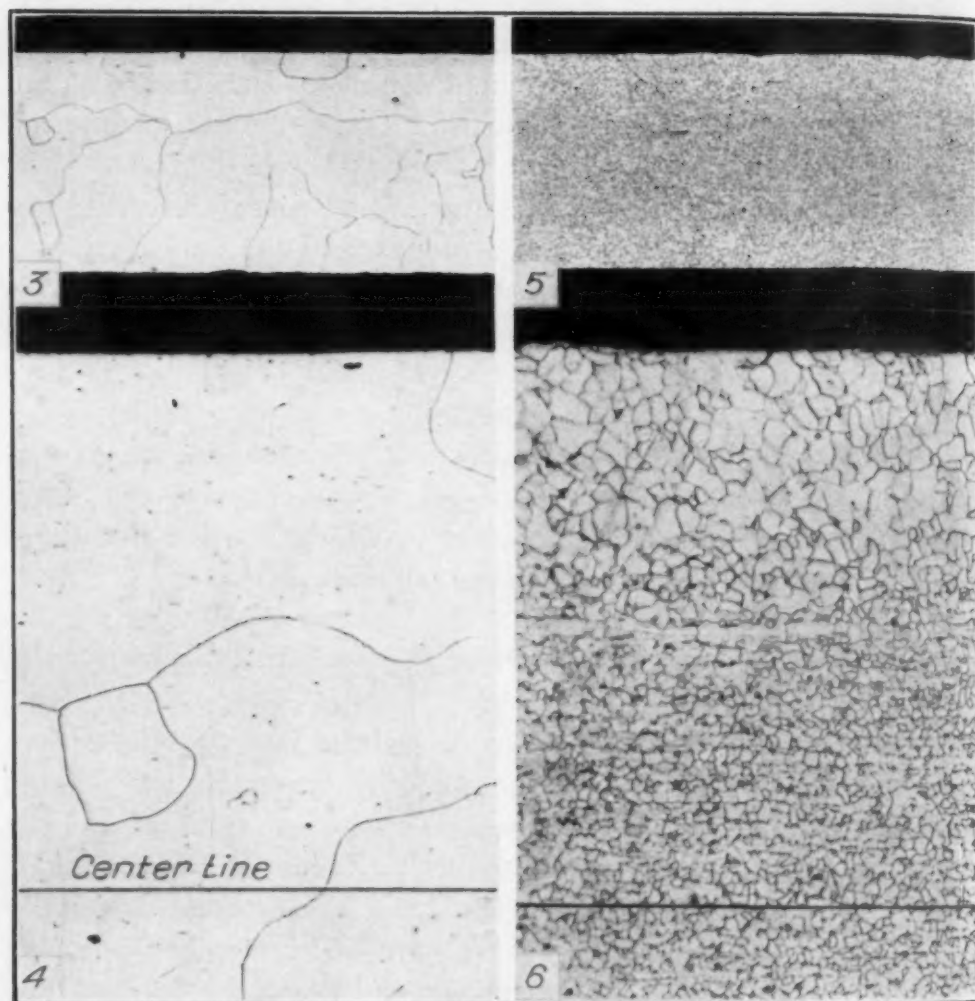


Fig. 3—Cross Section of Sheet Shown in Fig. 1.  $\times 20$ .  
 Fig. 4—Detail of Fig. 3.  $\times 100$ .  
 Fig. 5—Cross Section of Sheet Shown in Fig. 2.  $\times 20$ .  
 Fig. 6—Detail of Fig. 5.  $\times 100$ .

ever, occur in numerous instances in which the phosphorus content is considerably lower, running as low as 0.015 in the ladle analysis and 0.030 in the failure. In the examination of many hundreds of failures and in the carrying out of experiments wherein sheets rolled from the top and bottom bars of ingots were compared, the writers have invariably found that the sheets coming from the top or segregated portion of the ingot have a very fine-grained core similar to



that shown in Fig. 6. In view of the fact that most authors suggest that phosphorus causes brittleness, in part at least, by producing large grains,<sup>2</sup> the structures were hard to reconcile with theory. A lead to what we believe to be the correct explanation was obtained from some hand-mill rollers who, without knowing there was a difference between some sets of experimental bars, observed on more than one occasion that one group was hard to roll, stiff, and frequently required an extra pass to get the regulation sheet length.

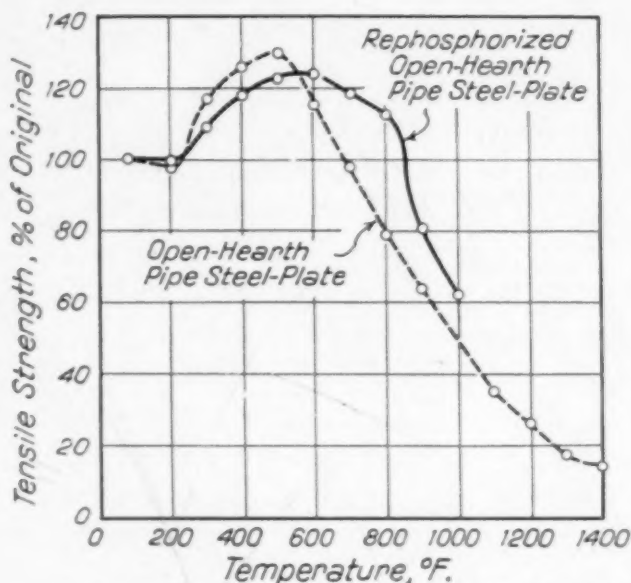


Fig. 7—Effect of Phosphorus on Tensile Strength at Elevated Temperatures. (Speller)

while the other group "slipped through" the rolls with ease. The fact that the pair which was most difficult to roll was from the top of the ingot led to the opinion that the higher phosphorus content was imparting greater resistance to deformation at the finishing temperature. Hence, for a given reduction, the residual strains from the cold-working effect would be greater in the segregated portion of the ingot and these sheets would recrystallize at a lower temperature upon subsequent box-annealing and therefore show a final grain size much smaller than would sheets similarly treated, but from that portion of the ingot in which phosphorus was not highly concentrated.

The particular lot of sheets from which these failures were taken was rolled without reheating from a bar having a 13.05 pound-

<sup>2</sup>"Alloys of Iron," Epstein, p. 353. Alloys of Iron Monographs.

foot weight to 18 gage and the pair furnace temperature was 1450 degrees Fahr. The sheets were, of course, finished at a black heat and the temperature was well within the range in which phosphorus imparts a marked stiffening influence, as is shown by Speller.<sup>3</sup> (Creep testing confirms the tensile test data.)<sup>4</sup>

The composition and physical properties of the steels which Speller was discussing were as follows:

	Chemical Composition Per Cent				Ultimate Strength Lb./Sq. In.	Yield Point Lb./Sq. In.	Elong. in 2" Per Cent	Red. Area Per Cent
	C	Mn	S	P				
Regular Open-Hearth	0.09	0.44	0.034	0.013	50,690	29,730	42.0	67.7
Rephosphorized Open-Hearth	0.09	0.43	0.050	0.103	61,120	38,785	37.0	63.8

It seems, therefore, that in the case of deep-drawing sheet steel the explanation for the way in which phosphorus produces low ductility is by causing too fine a grain. This explanation, of course, would have no relation whatever to the way in which a considerable quantity of phosphorus produces brittleness in hot-worked material.

This possible mechanism would explain why one sheet may show a hard, excessively fine-grained core while another sheet rolled with it as a pair and, therefore, receiving identical heat and mechanical treatment, may show a desirable grain of a much larger size. It also indicates why the relatively pure skin on a sheet coming from the upper portion of an ingot usually develops a larger grain size than the high phosphorus core, thus contributing noticeable irregularity as shown in Fig. 6. In considering the action of phosphorus in such a case, other elements which segregate along with it may also enter the picture. Hague and Brace report that sulphur increases the hardness of sheets to a noticeable extent.<sup>5</sup>

The difficulty of getting good drawing results from excessively fine-grained sheets has been pointed out by numerous investigators<sup>6, 7</sup> and in our experience we found this to be true. An equiaxed grain of approximately the size shown in Fig. 16 seems to be optimum, as this size produces little or no "orange peel" or surface roughening and at the same time affords a high degree of ductility.

<sup>3</sup>Discussion, *Proceedings*, American Society for Testing Materials, Vol. 24, 1924, Pt. II, p. 163.

<sup>4</sup>H. C. Cross and D. E. Krause, "Phosphorus As An Alloying Element in Steels for Use at Elevated Temperature," *Metals and Alloys*, Feb. 1937, p. 53-58.

<sup>5</sup>F. T. Hague and P. H. Brace, "Annealing and Normalizing Auto Body Steel in Electric Furnaces," *Iron and Steel Engineer*, September 1936, p. 47-58.

<sup>6</sup>Winlock and Kelly, "Sheet Steel and Strip for Automobile Bodies," *TRANSACTIONS*, American Society for Steel Treating, Vol. 18, 1930, p. 171.

<sup>7</sup>McGarity and Anderson, "Effect of Normalization on the Grain Structure and Physical Properties of Automobile Sheet Steel," *TRANSACTIONS*, American Society for Metals, Vol. 21, 1933, p. 142.

## SOURCE OF COARSE-GRAINED FAILURES

Referring to the "rotten break" type of failure shown in Fig. 1, the photomicrographs (Figs. 3 and 4) reveal clearly why the sheet failed to form. This is an extreme case, of course, but rejections are met all along the series from a grain size just large enough to develop "orange peel" upon deep-drawing to the actual breaks as shown above.

In an effort to eliminate loss of material from the above-mentioned trouble, it will simplify matters if the mill operator will recognize the importance of the relation between annealing temperatures and the previous amount of cold work done on the sheets. Since the annealing cycle cannot be juggled readily to meet various degrees of strain in the sheets, a closer study of the cold-working factor is in order.

It is well established that for increasingly severe cold-working the temperature at which the metal will recrystallize becomes lower and that as the recrystallization temperature is lowered there is a corresponding decrease in grain size. This thought carries the picture of varying hot-mill finishing temperatures to most operators, and justly so. There are certain other factors, however, which influence the amount of strain produced by a given reduction at a given temperature.

An interesting experiment was made in which the effect of the grain size of the sheet bar or strip, previous to rolling, was studied. Fig. 8 shows the experimental procedure. Hot-rolled  $\frac{3}{16}$ -inch strip was selected and cut into two parts, insuring uniformity of composition in the two sections. Millings from the full cross section of this strip showed the following composition:

	Per Cent
Carbon	0.06
Manganese	0.36
Phosphorus	0.020
Sulphur	0.014
Silicon	0.010

One part (No. 2) was given a box-anneal at 1350 degrees Fahr. which produced enormous grain growth, as is shown in Fig. 13. The other piece of strip (No. 1) was held without treatment and had the grain size shown in Figs. 9 and 12. The two pieces were matched as a pair and rolled from one heating into 18-gage sheets. The finished



sheets were box-annealed at 1450 degrees Fahr., the sheets not being opened up before annealing. The grain size of the sheet from the fine-grained strip is shown in Figs. 11 and 16 and the grain size of the sheet from the coarse-grained strip is shown in Figs. 15 and 17.

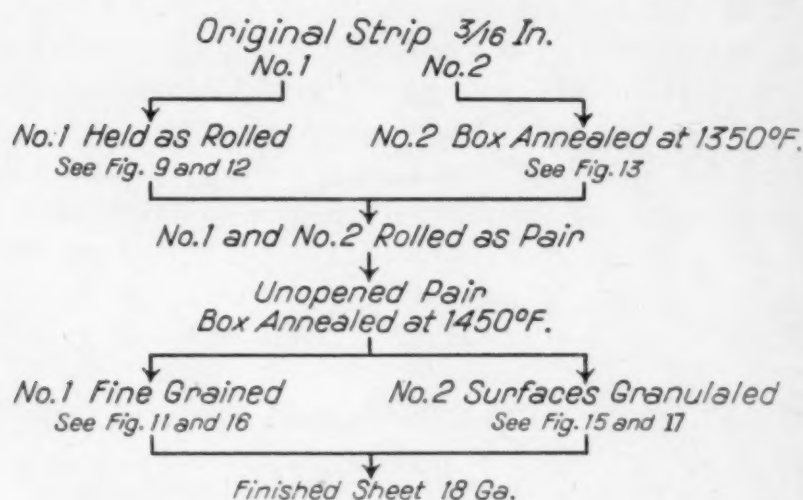


Fig. 8—Procedure Used in Rolling Experiment.

Another pair of fine- and coarse-grained strips was treated in a similar manner, except that rolling was stopped in the breakdown condition so as to give an intermediate stage. The breakdowns were annealed along with the finished sheets with the results shown in Figs. 10 and 14.

In both the breakdown stage and in the finished sheets from the coarse-grained strip (Figs. 14 and 15) one can trace the effect of grain size previous to rolling. The surfaces of the breakdown and finished sheet show abnormal grain growth, while the centers, coming from the fine-grained center of the original strip (Fig. 13), have a normal box-annealed grain size.

In this experiment the difference in grain size in the strip prior to rolling was carried to an extreme never reached in practice, but it shows quite clearly that the grain size of the sheet bar has a marked bearing upon the grain size of the finished sheet. Microsections of the 18-gage sheets as rolled showed no well-defined grain boundaries in either of the sheets previous to box-annealing. Both the sheet from the coarse-grained and from the fine-grained strips had the usual, completely fibrous, cold-worked structure. It is evident from this that the excessively large grains in the strip do not persist unchanged through the rolling operation, but that they influ-



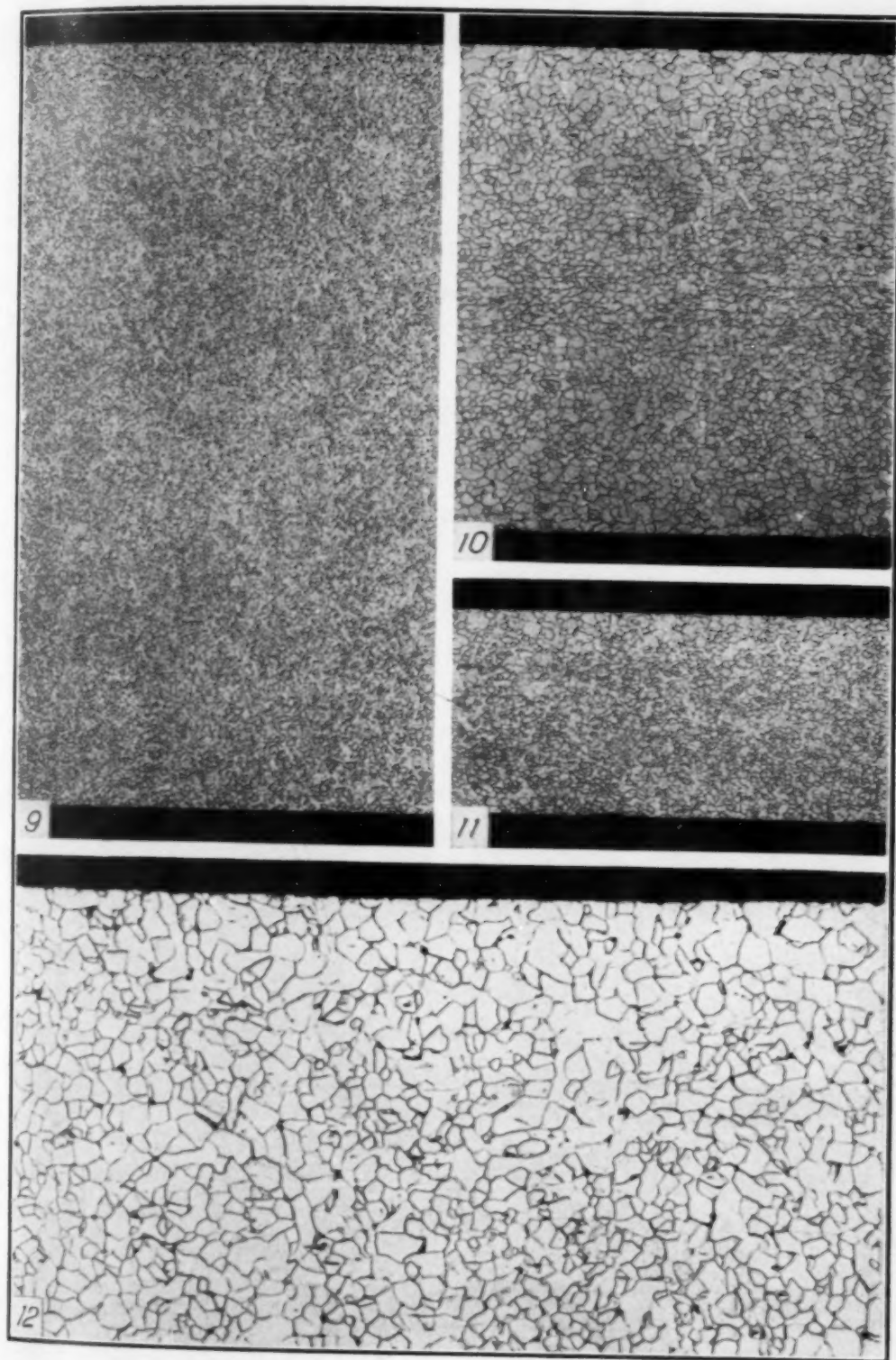


Fig. 9—Structure of Hot-Rolled Strip—Longitudinal Section.  $\times 20$ . Millings from Surface Ran 0.06 Per Cent Carbon.

Fig. 10—Breakdown from Strip in Fig. 9. Box Anneal, 1450° Fahr.  $\times 20$ .

Fig. 11—18-Gage Sheet from Strip Shown in Fig. 10. Box Annealed at 1450 Degrees Fahr.  $\times 20$ .

Fig. 12—Detail of Structure in Fig. 9.  $\times 100$ .

ence the degree of strain produced by rolling and, hence, have a marked bearing upon the grain size developed in the finished sheet by recrystallization during box-annealing.

It is the practice in most mills to stack the long sheet bars hot as they come from the finishing stand. Usually high pressure water sprays are played on the rapidly moving bar just before it reaches the last roll in order to crack off the scale and to cool down the bar

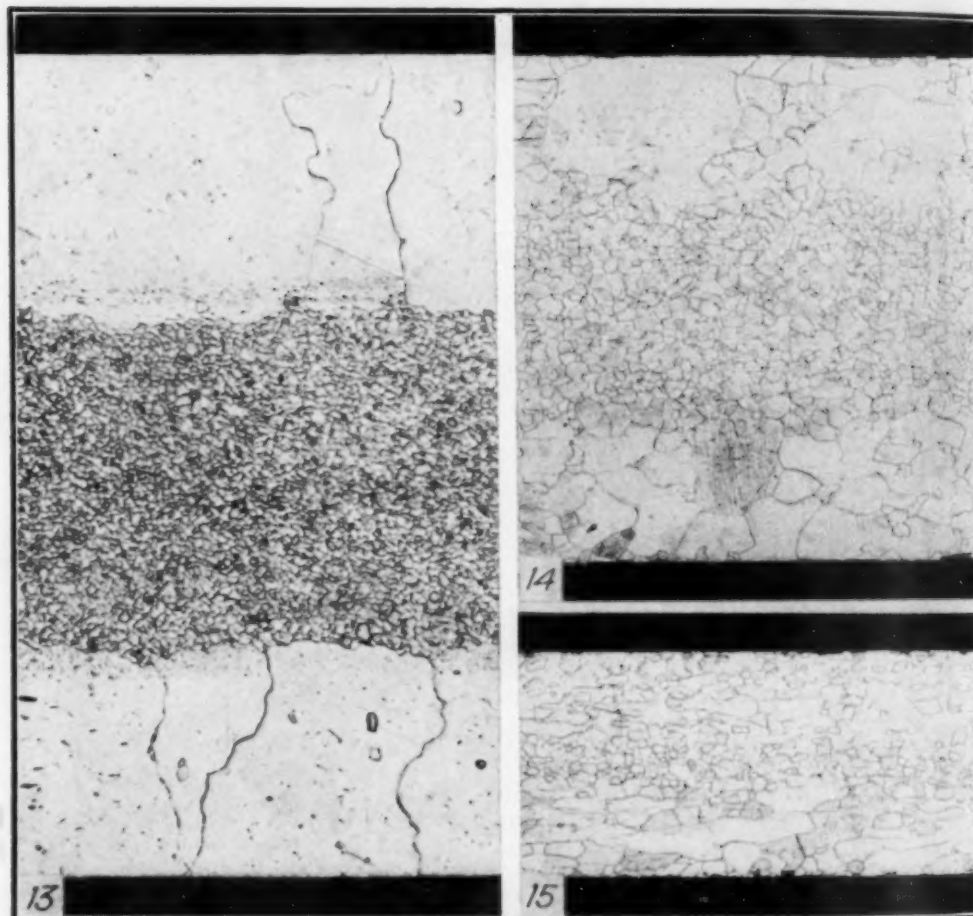


Fig. 13—Hot-Rolled Strip Box-Annealed at 1350 Degrees Fahr.  $\times 20$ . Millings from Surface Ran 0.04 Per Cent Carbon After Box Annealing.

Fig. 14—Breakdown from Strip Shown in Fig. 13 Box-Annealed at 1450 Degrees Fahr.  $\times 20$ .

Fig. 15—18-Gage Sheet Rolled from Strip Shown in Fig. 14. Box-Annealed at 1450 Degrees Fahr.  $\times 20$ .

so that a thick layer of scale will not form after finishing. The water cooling and pressure from the finishing rolls leave the surface of the bar with varying degrees of strain, depending upon the temperature of the steel. Flying shears cut the hot bar into lengths, ranging around 30 feet, and these long bars come out successively

upon an automatic table which drops the thickness of a bar with each one. The bars from as many as three ingots are usually stacked in one pile on the table before removal by an overhead crane. The stack of hot bars may reach a height of three feet, depending upon the width of the bars being rolled, and the heat from the interior of the bars holds the temperature of the stack high enough and for long enough time to permit considerable grain growth on the surfaces and edges of the bars. Such growth is shown in Fig. 18.

The pair furnace temperature is well below the  $A_{c3}$  point for steel of this composition and, hence, the grain of the bar, whether it be large or small, persists through the pair furnace to the hot mills where it exerts a noticeable effect upon the amount of work or energy necessary to produce a given reduction. The significant point here is that, other factors being the same (pair furnace temperature and amount of reduction on the hot mill), the sheet coming from a fine-grained bar will be more severely strained than one coming from a bar such as that shown in Fig. 18 and, hence, will recrystallize at a lower temperature and have, therefore, a finer grain after box-annealing.

In addition to the relationship between cold-working, recrystallization temperature, and grain size (which is common to nonferrous metals as well), the sheet manufacturer gets coarse-grained failures from the so-called abnormal grain growth. Sauveur discovered in 1912 that in low carbon steels there is a "critical degree of strain" which produces abnormal grain growth upon subsequent slow annealing. This phenomenon has caused the sheet producer and consumer an enormous amount of trouble. The usual remedy is to cold work the sheets as severely as possible so as to overstrain the material, in which condition abnormal grain growth is not so prevalent. This remedy does not always solve the problem, however, especially when high box-annealing temperatures are used. The mechanism of this abnormal grain growth, which caused the failure shown in Fig. 1, will not be discussed here, not because it is unimportant but because it has been so clearly and completely covered by other investigators.<sup>6</sup>

It is quite possible to reclaim lots of sheets which have been rejected on account of excessive grain growth by normalizing and re-box-annealing the material, provided slightly higher normalizing temperatures are used or slower speeds through the normalizing furnace can be adopted. The sheets showing abnormal grain growth are usually badly decarburized and this causes a sluggish reaction



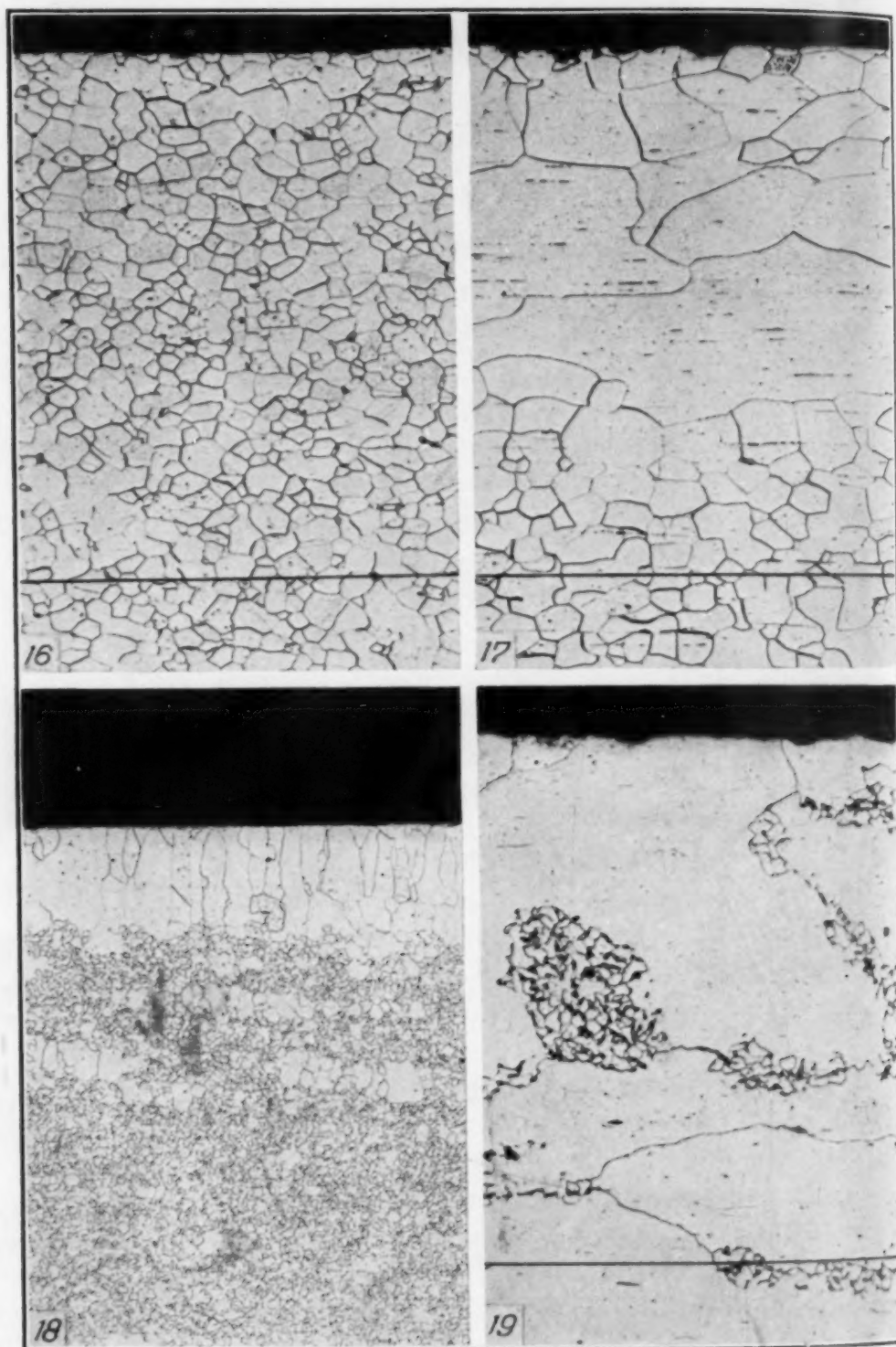


Fig. 16—Detail of Fig. 11. Structure of the Sheet Rolled from the Fine-Grained Strip.  $\times 100$ .

Fig. 17—Detail of Fig. 15. Structure of the Sheet Rolled from the Coarse-Grained Strip.  $\times 100$ .

Fig. 18—Sheet Bar Stacked Hot.  $\times 20$ .

Fig. 19—Coarse-Grained Sheet Imperfectly Normalized.  $\times 100$ .



in the normalizing furnace, an example of which is shown in Fig. 19.

In box-annealing normalized sheets the temperatures have to be held below those used in annealing sheets directly from the hot mills on account of greater susceptibility to grain growth of the abnormal type. This necessarily low box-annealing temperature varies with the speed of cooling from the normalizing temperature in a very interesting way and is apparently due to a critical degree of thermal strain produced by cooling through the transformation range.<sup>8</sup>

#### TESTING AND THE DEPENDABILITY OF TEST RESULTS

Doubtless, no one particular test is best for all purposes. The combination of tensile tests and microsections for grain size is exceedingly good. A sheet having a uniformly large grain size may give excellent tensile results, but the microscope would indicate at once that surface roughening would take place if the forming were severe. On the other hand, a sheet may have an excessive amount of cold-rolling after annealing and yet show no slip bands in the ferrite grains which may be equiaxed and of a desirable size, but the tensile test immediately shows a high yield point and comparatively low elongation values. The tensile test is of great value in indicating whether or not the sheet is subject to stretcher strains, as shown by the shape of the stress-strain diagram around the yield point. There are, of course, very difficult drawing operations in which one steel will just fall short and in which another will just pass the requirements. The difference between the good and the bad, in such cases, is so slight that it may be caused by a small gage variation, surface finish, or something aside from the physical properties of the steel and no test can reasonably be expected to make an exact differentiation. The important point here is that, in almost any small mill producing sheets for moderately deep drawing, large variations in the quality of the finished material may be found. The dependability of existing testing apparatus far exceeds the ability of the producer to control all the factors governing the uniformity of his product.

The physical testing of sheet material for cold-forming has been a subject of much discussion and debate. Producers have reported the filling of orders, with satisfactory results, from material which

<sup>8</sup>Kinsky. "The Formation of Columnar Crystals in Steel Sheets After Normalizing," *Journal, Iron and Steel Institute*, Vol. CXXVII, No. 1, 1933.

had been rejected by another customer for making the identical article.<sup>9</sup> This convinces such a producer that most of the trouble with failures is due to improper dies or something else traceable to the consumer's practice. The writers once knew a mill superintendent who became so disgusted with continual failures from material approved by the metallurgical department that he shipped out a lot of sheets rejected by the metallurgical inspectors as unfit for deep-drawing. The said superintendent, after assuming the responsibility of the shipment, was gratified a few weeks later when he received a letter from the customer praising the "rejected" material and asking for more sheets of the same type.

This endless confusion has given rise to some question concerning the reliability of various means of testing and, in many cases, of the unfortunate men who are making the tests. A review of the variables which enter into the testing program might throw some light upon the meaning of the tests.

In the first place, there is a very marked difference between the ductility of sheets coming from the bottom and from the top of a given ingot and there is also some difference between the ingots in the same heat. No two heats behave exactly alike and the processing factors in the sheet mill may vary markedly from one pair of sheets to another. Usually the ingots are kept in order through the bar mill, but from that point on there is hopeless confusion. Sometimes as many as twenty different orders may be filled from one heat or one order may have parts of several heats in it. At the bar shears the identity of location in the ingot is completely lost as four bars may be sheared at once, each coming from unknown parts of different ingots. Further shuffling occurs in loading the pair furnaces and when the test sheets are taken from certain locations in the stack after box-annealing their reaction to physical tests depends, among other things, upon the following variables:

1. Composition of the steel.
2. Location in the ingot from which the sheet bar comes.
3. Grain size of the bar or strip from which the sheet was rolled.
4. Finishing temperature on the hot mill.
5. The box-annealing cycle.

The chance factor regarding the location in the ingot of any particular sheet taken for testing may far outweigh small variations

<sup>9</sup>H. W. Gillett, "Testing Deep-Drawing Qualities of Sheet Metal," *Metals and Alloys*, Oct. 1931, p. 217-222.

in different annealing cycles. It can easily be seen that a lift of good sheets may be rejected because a bad sheet happened to be taken for a test and, on the other hand, a bad lot of material may be passed because one or two test sheets happened to be good.

These difficulties are easier to point out than to correct. Obviously, the complete identification of each sheet would be well nigh impossible on a production schedule. Some producers have even resorted to the practice of using only bars from the lower portion of the ingots for especially difficult deep-drawing orders. In the case of some of the large strip mills they regularly route slabs from the top portion of their ingots to lower grade products, using only the material from the central and lower portions of the large ingots for deep-drawing stock. The identification of the different coils as to ingot location is carried through by stamping a code number on the strip and this simplifies the testing problem because the routine tests can be taken from the poorest material, thereby eliminating the chance factor.

Segregation of material at the bar mill into lots coming from the upper and lower parts of the ingot, in a manner somewhat similar to the A, B, and C divisions of rail steel, would no doubt be feasible were it not for complications arising from differences in foot weight requirements for the various orders. The gage and length of the sheet, of course, determine the foot weight of the bar and seldom are two orders the same as regards this point. If the hand-mill operator could divide his bars in this manner and roll the bars from the top portion of the ingots into roofing, siding, or some material which requires little or no cold-forming and those from the lower or least segregated portion into material for deep-drawing, a decided improvement would result. There is an increasing demand for greater uniformity of material and this is being met by continuous strip mills. Unless some step such as this can be taken, it seems likely that the product of the hand mills will not stand up in competition with that of the continuous mills except for those uses in which the poorest sheets of the lot are adequate.

#### ACKNOWLEDGMENTS

The authors take this opportunity to express their indebtedness to Mr. Thomas Conway of the Tennessee Coal, Iron and Railroad Company for much aid in the preparation of the paper.



## DISCUSSION

**Written Discussion:** By A. L. Davis, research engineer, Scovill Manufacturing Co., Waterbury, Conn.

This paper by metallurgists of the Battelle Memorial Institute, is of the excellence we have grown accustomed to expect from that source. The authors are to be congratulated. Their interpretation of the fine grain in the segregated cores of sheets as being due to the greater resistance of such cores to reduction during finish rolling (at a black heat), with resulting greater strain, and hence recrystallization at a lower heat when annealed is entirely logical. We all are familiar with the fact of finer grain resulting from more severe cold work before annealing, provided the annealing temperature be not too high. However it is also true that the higher content of the core in metalloids also tends to obstruct grain growth.

I check the grain size indicated by their Fig. 16, as an optimum for deep drawing with little or no orange peel and yet high ductility, judging its grain size to be about No. 7 on A.S.T.M. chart. Actually we regard 0.022 to 0.032 millimeter as the best range.

Their interesting experiment in processing, to intentionally develop abnormally coarse grain on sheet surfaces (Fig. 14) is instructive. Doubtless it will cause nods of recognition from all steel producers. Critical strain and annealing temperatures can cause a lot of evil pranks by the goblins in the metal.

The remarks as to testing, and the causes of trouble in deep drawing steel sheets, apply to cold rolled steel strip as well. Engineers should certainly believe in the value of tests.

It is vastly important, however, to test a sufficient number of samples to assure that we have a fair run of the metal in question. Having given such an adequate sampling, I firmly believe "test" results are more likely to be right than is the usual so-called practical test at the press, (which may be superficial), where correct adjustment of the blank holder, proper design and setting of tools and their lubrication, are of equal importance with the quality of the steel, in obtaining success.

Admittedly no single laboratory test subjects sheet metal to the combination of deformational stresses which are present in the drawing operation. Hence, no one test is an infallible criterion of performance in drawing. On the other hand, a combination of tests, including analysis, hardness, ductility, grain size, microstructure and surface finish, do definitely determine the properties of the metal, and afford an accurate measure of its drawing capacity—under given conditions.

It is not practical to make all the above tests as a matter of routine, nor is it necessary. When metal is produced under close analytical control of raw material, and standardized and controlled practice in melting, rolling, re-rolling, finishing and annealing, the need is for a voluminous running check on hardness and approximate grain size. The ability to make a great number of these tests quickly and cheaply offsets minor lack in their precision.

As a consumer, I heartily endorse the practice reported by the authors of separating the bottom portion of the ingot and applying it to difficult jobs;

leaving the upper (segregated) portion of each ingot for less exacting uses in production.

**Written Discussion:** By O. E. Romig, assistant research engineer, Carnegie-Illinois Steel Corporation, Research Laboratory, Pittsburgh.

We desire to discuss particularly the authors' conclusions concerning the type of failure shown in Fig. 2. The authors state that this failure is "the result either of poor open-hearth practice or improper shear discard." We doubt whether either of these conclusions is a major factor. The photomicrographs in Figs. 5 and 6 do not reveal unsound steel, and it would be illogical to expect a steel manufacturer to crop-shear and discard steel of the quality shown. The chemical and physical tests exhibited in Table I do indicate phosphorus segregation, and it is perhaps reasonable to conclude that the phosphorus content was higher than advisable for the intended job. The effect of phosphorus as a hardening element is well known, and this punched edge must not be too hard if it is to draw without cracking. It is common knowledge, we believe, that difficulties are generally more prevalent in drawing operations where an opening is punched before the forming takes place. The cleanness with which this punching is done, freedom from burr and the minimizing of strain due to same is an important factor. With this cold-worked, punched edge being high in phosphorus, its hardness is probably increased with consequent greater danger of failure. We have observed cases where filing or grinding to smooth the punched edge helped to eliminate this type of cracking. Also torch annealing the punched edge or pot annealing the punched blanks has removed punching strains and lessened breakage in actual practice. Since the fractures were not limited to one direction but occurred at random around the circular opening, under annealing is not, in our opinion, an indicated cause of failure, although a higher temperature might have resulted in some grain growth and consequently a softer sheet but perhaps not without danger of excessive grain growth to other sheets in the box.

The authors' explanation of the manner in which phosphorus produces low ductility, by causing a fine grain, appears logical. However, the fact that phosphorus raises the  $A_{c1}$  of iron may be worthy of mention. (This is described by Osmond, *Journal, Iron & Steel Institute*, 37, No. 1, 38, 1890—H. M. Howe, *Iron, Steel and Other Alloys*, New York, 1906; Howe and Levy, *Transactions of the American Institute of Mining and Metallurgical Engineers*, 47, 603, 1913—Haughton, *Journal, Iron & Steel Institute*, 1927, No. 1, p. 417, and Abbott, *Iron Age*, 1933, Vol. 132, December 28, p. 7-11). This, of course, relates to the previous history in production from ingot form where the higher  $A_{c1}$  point might produce an effect on grain size. Phosphorus also raises the  $A_{c1}$  point (Reported by Kjerrman, *Transactions of American Society for Steel Treating*, March 26, Vol. 9, p. 430-451) which would account for poorer response to annealing since probably the steel at the surface of the sheet is quite as severely worked by rolling as the inner portion or core. These factors may aid in explaining the smaller grain and cored structure exhibited in Fig. 6 and be at least contributory factors to the smaller grain of the central portion, richer in phosphorus, as shown in that photomicrograph.

The authors are to be complimented on the clear presentation of the factors and related problems affecting finished grain size of noncontinuous mill

products. Their citation of the difficulties in metallurgical control on hand mills is appreciated. The feature of surface is also an important problem in the production of a uniform product from such mills. It is true that competition from continuous mills is severe, but as stated by the authors, the flexibility and ability to fill quickly and economically small orders, will probably enable non-continuous mills to hold a place in the industry for some considerable time.

#### Oral Discussion

A. L. DAVIS:<sup>1</sup> I can corroborate the statement of the last commentator, to the effect that when a pierced hole is drifted out in a steel sheet, the condition of the pierced edge is a very important factor. Frequently reaming the edge, or otherwise improving its condition, will solve the trouble, and bring success where failure has been the previous experience.

#### Authors' Reply

The written discussions submitted by Messrs. Davis and Romig represent, by accident, the views of both the consumer and the producer of sheet steel.

Mr. Davis' substantiation of our results as regards the grain size which is optimum for deep drawing is reassuring in view of his long experience in the testing and use of sheet material. It is recognized, of course, that for certain applications a much finer grain may be desirable as, for example, where the smoothest possible surface is wanted for plating without preliminary buffing. The grain size, as shown in Fig. 16, was simply said to be best so far as ductility was concerned.

It is also of interest to note that Mr. Davis places considerable reliance in standard tests as compared to the results at the forming press. His explanation of this point seems entirely adequate.

In attempting to explain the cause of failure, it is usually found that the customer thinks the manufacturer of the sheets is responsible for breakage and, conversely, the steel maker believes the cold forming practice or equipment is at fault. Mr. Romig, we think, has been trained in this line of reasoning and is simply upholding the sheet producer's side of the question. He doubts that the open-hearth practice had anything to do with causing the failure shown in Fig. 2, placing the responsibility on the barrel maker for not observing the following precautions:

1. Filing to smooth the punched edges before pressing.
2. Grinding to smooth the punched edges before pressing.
3. Torch annealing to remove punching strains before pressing.

These openings, of course, are stamped and pressed for threaded fittings in one operation. A satisfactory head could no doubt have been made from this sheet if the barrel manufacturer had followed the precautions suggested by Mr. Romig, but a simpler solution for him was to buy sheets which would form under the conditions obtaining in his plant. Steel barrel manufacture is carried out on a mass production basis, as are most cold-pressing operations, and intermediate steps or treatments are avoided where possible. One barrel maker<sup>2</sup>

<sup>1</sup>Research Engineer, Scovill Manufacturing Co., Waterbury, Conn.

<sup>2</sup>Steel Barrels from Flat Sheets in One Hour, *Iron Age*, Vol. 129, p. 876-878, April 14, 1932.



advertises the fact that within one hour after receiving an order he can have a full truck load of drums ready for shipment, starting with flat sheets, and this includes a twenty-minute period for the paint to dry. Instead of resorting to the above-mentioned precautions to prevent breakage, the barrel maker would suggest that the steel maker stand the additional expense of further refining his steel in the open-hearth before tapping.

The effect of the high phosphorus content in the sheet (0.076 per cent) is reflected in the physical properties, as can be seen in Table I. The yield strength of 43,000 pounds per square inch is far too high, and the elongation—31 per cent in two inches—is too low. In view of these figures and the distinct core effect as shown in Fig. 6, we still maintain that, for the purpose intended, the steel maker is at fault for this failure. It is stated on the sixth page that this heat should have been diverted to a lower grade product. Roofing or siding made from this steel would have served equally as well as when made from truly deep drawing quality sheets. This point, of course, only justifies extensive discussion, because it is representative of scores of similar arguments which arise daily. An effort was made throughout this paper to show that the steel maker must carefully consider the requirements of the individual orders and control his product according to these needs.

In view of Mr. Romig's tolerant attitude towards phosphorus, we might add that one manufacturer of cold-reduced strip for automobile bodies places a limit for fender stock at 0.010 per cent phosphorus. Those heats showing 0.011 per cent phosphorus are, assuredly, not scrapped but are diverted to orders calling for slightly less deep drawing.

RELATION OF SIZE OF SPHEROIDS IN TOOL STEEL  
TO ITS MACHINABILITY  
AND  
TO HOLDING EDGE OF CUTTER

BY DONALD E. RODA

*Abstract*

*The quality of a machined surface depends, among other factors, on the ability of the cutter to hold its sharp cutting edge. This paper illustrates how a small difference in the size of carbide particles in a tool steel makes a great deal of difference in fracturing the cutting edges of cutters.*

*A new method of breaking down the very stable iron and manganese carbides in steel is stated.*

*The microcharacter was used to show definitely the characteristics and well appreciated hardness of the carbide spheroids in spheroidized steel.*

IN a small room adjoining a young, energetic manufacturing plant, a skilled tool maker devotes his entire time to engraving type matrixes with which type are rolled. Though his troubles are small and not exactly duplicated in this large manufacturing world, his experiences and task are well suited for the study of the wide spread influence of spheroidized carbide particle size on quality of machined surfaces and tool life between grinds. In contrast to thousands of parts between sharpening with some screw machine tools, the tool life in this problem is limited to the cutting of the matrix for one or two typewriter characters. The type must have a flat surface with sharp corners to produce well defined letters. A line width of 0.0015 inch and thus a similar flat in the bottom of the type matrix, would be desirable in some cases such as small elite type, the engraver has found, however, that 0.0025 inch is the narrowest face that he can consistently machine. Even then, he encounters shipments of steel that are almost impossible to use.

A face width of 0.0025 inch and a depth of 0.025 inch define

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A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. The author, Donald E. Roda, is metallurgist, Electric Writing Machine Division of International Business Machines Corp., Rochester, N. Y. Manuscript received June 21, 1937.

the matrix which is rough cut 0.021 inch deep and finish cut 0.004 inch deeper. With smooth side walls, sharp corners, and a flat bottom, the matrix will produce good type and eventually fine typing. When the point or cutting edge of the engraving cutter breaks or chips, the letter must be cut deeper and an equal amount taken off of the top of the matrix to maintain the depth at 0.025 inch. Some lots of steel are difficult to machine to these specifications, while the cutters easily machine two or three letters with other lots of steel. The following table does not indicate the degree of difficulty in machining the hard steel; it only indicates the maximum number of letters that can be engraved with the hard and soft steel before the cutter has to be resharpened.

Maximum Number of Characters Cut Between Cutter Grinds

	Hard Steel	Soft Steel	Hard Steel Annealed
Rough Cut .....	2	4	5
Finish Cut .....	1	2	2

The steel used for the type matrix is an oil hardening die steel of the manganese type with an approximate composition of 0.90 per cent carbon, 1.55 per cent manganese, 0.30 per cent molybdenum. Since one steel has been designated as hard because it was hard to machine, and the other soft because it was easy to machine, it must not be assumed that there is any difference in the hardness as usually designated, for the Rockwell hardness readings overlapped and an average of several readings indicated only a difference of two Rockwell B points.

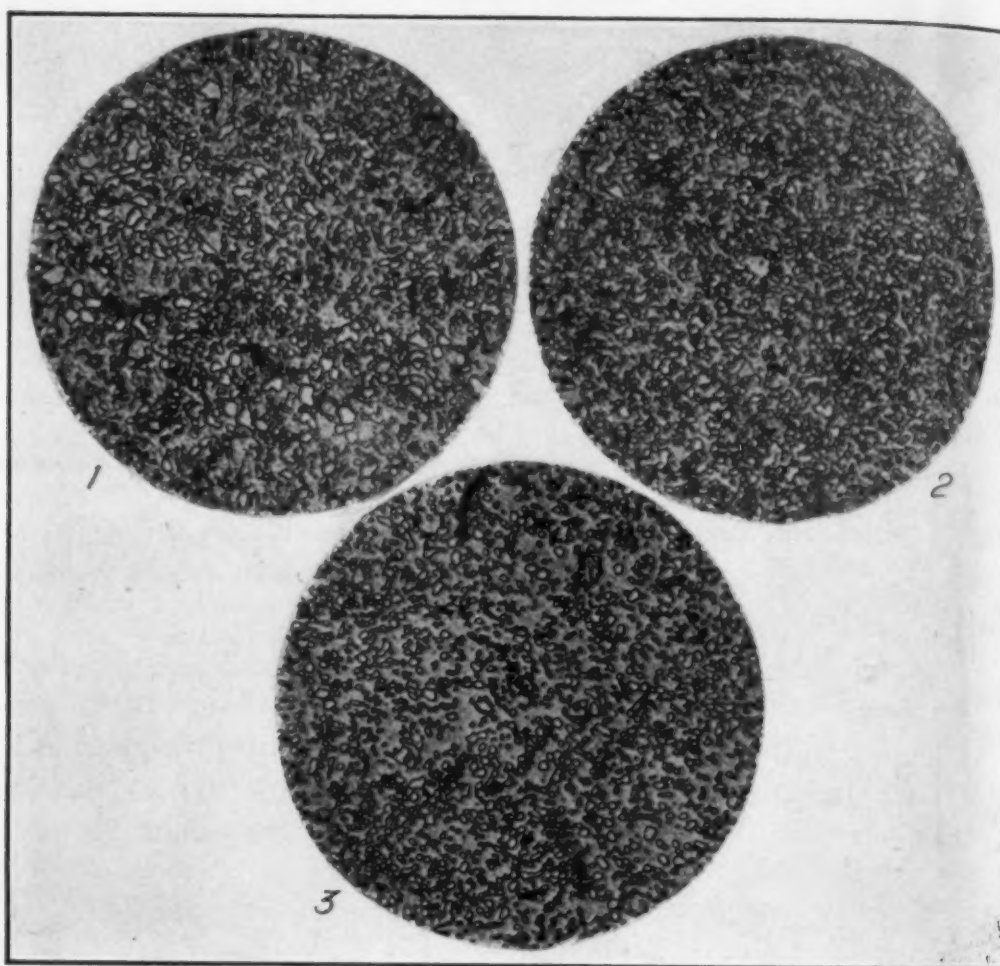
Rockwell Hardness Readings

Soft		Hard		Hard Annealed	
45-T		45-T		45-T	
58.5, 59	} 59	60, 60	} 60.5	59, 59	} 59
59, 56		60, 65.5		59.5, 60	
61, 59		61, 61		58.5, 60	
or	B 85.5		B87.5		B 85.5

Upon microscopic examination to find the reason for the great difference between a hard and soft lot of steel a few slightly larger carbide particles in the hard sample than those in the soft sample was the only explanation found. This can easily be seen by comparing photomicrographs Fig. 1 and Fig. 2 respectively.

Both pictures represent good examples of commercially spheroidized tool steel. It was decided that the large carbide particles would have to be broken down to save the sharp corners of the engraving cutter, and it was found, contrary to popular belief, that this took place when the steel was annealed at 1250 degrees Fahr. Photomicrograph Fig. 3 is of a  $\frac{1}{2}$  inch thick sample taken adjacent to





Figs. 1 to 3—Photomicrographs Showing the Spheroids in Specimens of Tool Steel.  $\times 500$ . Fig. 1—Structure of Tool Steel Difficult to Engrave. Fig. 2—Structure of Tool Steel Easy to Engrave. Fig. 3—Annealed Structure of Tool Steel Easy to Engrave.

Fig. 1 and annealed at 1250 degrees Fahr. for three hours, cooled over night in the furnace to 800 degrees Fahr. and then air-cooled. The steel was covered with clean cast iron chips to prevent scaling. The large carbide particles broke down and all of the particles became more rounded. In order to ascertain more accurately than by mere observation of the photomicrographs what happened to the steel, the pictures were enlarged to approximately a magnification of 1500 diameters and the number of carbide particles in a  $4\frac{1}{2}$  inch circle was determined. There were 865 particles in the steel that was hard to machine, 980 spheroids in the steel that was easy to machine, and 1180 in the hard steel after annealing, as would be expected; smaller spheroidized carbides and more of them. Similarly the largest carbide particle sizes varied from 0.0002 to 0.00015 to 0.00011

inch. The 0.0002 inch particles in the hard steel appear in groups while the smaller spheroids are more uniformly distributed. A 0.0002 inch obstacle to a 0.002 inch cutter point might not be a serious objection, but a cluster of such particles in the path of the cutting edge might offer too great a resistance to be machined.

Although the experiment was carried out on a large scale, the engraver only needed to cut one letter in the annealed steel to tell

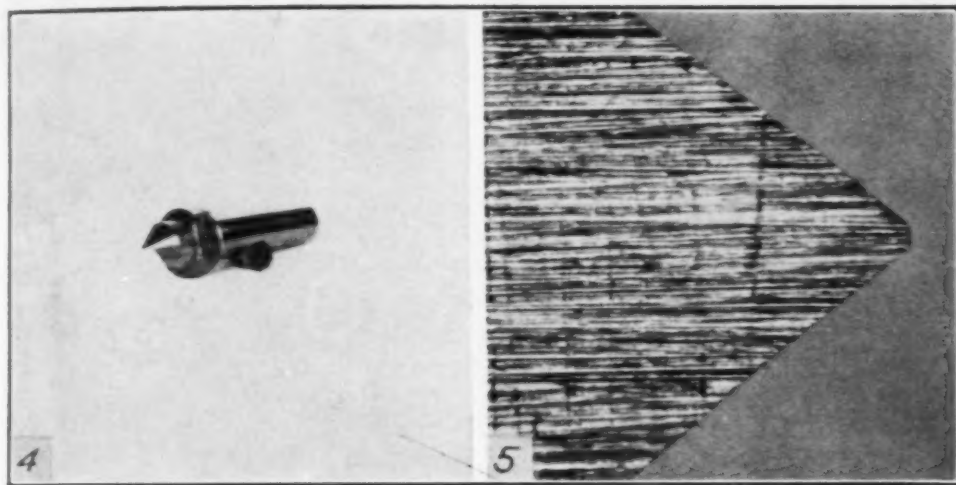


Fig. 4—Photograph of the Cutter Used in These Tests Slightly Less than Full Size. This Cutter is Used to Shape the Die Impression.

Fig. 5—Photomicrograph of Cutter Point After Grinding with a Flat on the End 0.0019 Inch in Diameter.  $\times 50$

that it was easy to machine. He did not believe that it was better than the soft steel, but it was certainly as good as any lot of steel that he had ever had. There was practically no change in the Rockwell hardness of the steel in the annealing operation as may be noted in the previous table; the hardness dropping two points to the same reading as the soft steel. Also, the maximum number of letters per grind increased to the same value or slightly more than could be cut in the soft steel.

Fig. 4 is the photograph of the cutter slightly less than full size, which is used to shape the die impression. It is a half cone with a 0.00125 to 0.002 inch flat on the point. The cutters are of high speed steel and are made in Germany. Fig. 5 shows a 50 diameter picture of the cutter point after grinding with a flat on the end 0.0019 inch in diameter. In sharpening, the cutter is ground on the surface of the cone much finer than is seen on the flat section through the center of the cone.

Fig. 6 is a photograph of the cutter after engraving one single

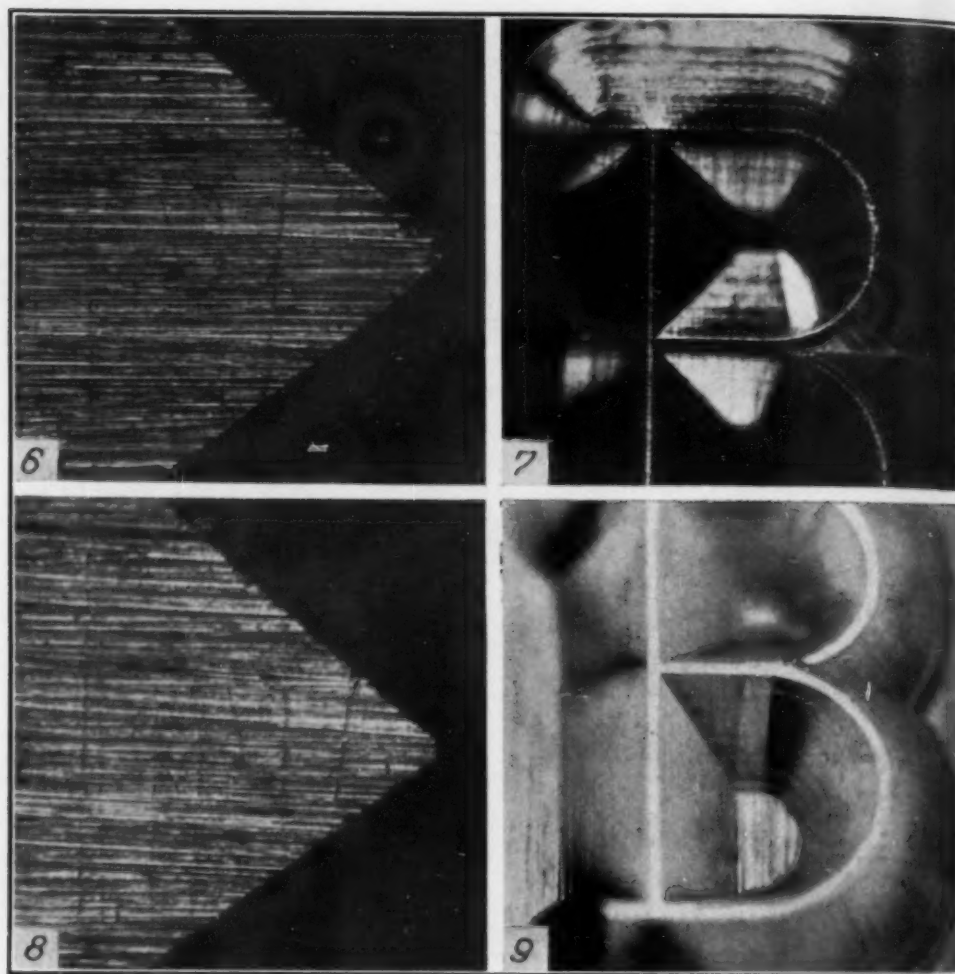


Fig. 6—Photomicrograph of Cutter After Engraving One Single Letter "B" in Hard Steel.  $\times 50$ .

Fig. 7—Showing the Effect of Broken Point on Cutting Tool Which Leaves Ridges in the Bottom of the Matrix.  $\times 25$ .

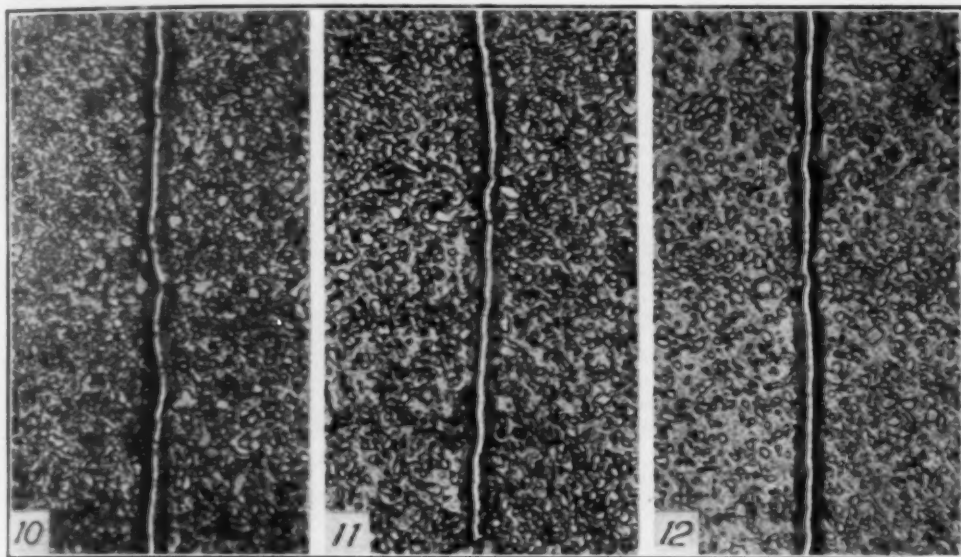
Fig. 8—Photomicrograph of Tip of Cutter After Cutting Annealed Steel. The Cutter is as Sharp After Making the Cut as it was Prior to Making the Cut. It Had a Flat of 0.00125 Inch in Diameter on the Point.  $\times 50$ .

Fig. 9—Matrix Cut by Tool in Fig. 8.  $\times 25$ .

letter "B" in the hard steel. The point is badly broken and could not be used to rough cut another letter. Since the rough cut is most destructive on the cutter, the pictures are all of rough cuts which may or may not be 0.0025 inch wide at the bottom of the cut. The cutters were ground very carefully, however, to observe how the sharp corners would stand up. The effect of the broken point is seen in Fig. 7 as ridges in the bottom of the matrix, and lines on the side walls indicate that the cutting edge was chipped. If the finish cutter does not stand up better than this, the engraver will have a just cause for complaint.



Figs. 8 and 9 show some almost unbelievable machining in the annealed sample of the hard steel. The photograph shows the cutter as sharp as before it engraved the letter, with a flat of 0.00125 inch (one and one-quarter thousandths of an inch) in diameter on its point which produced a face 0.0019 inch wide in the bottom of the matrix. This probably explains why a face of 0.0015 inch cannot be



Figs. 10 to 12—Bierbaum Microcharacter Cuts Made on Three Specimens of Tool Steels. Fig. 10—Hard Steel. Fig. 11—Soft Steel. Fig. 12—Annealed Steel.  $\times 500$ .

obtained for the cutter cuts large, and a point sharp enough to produce 0.0015 inch on the bottom would not stand up. The flat bottom, sharp corners, and smooth side walls are well brought out in Fig. 9 that was taken at an angle to the face of the die. This letter was cut in the same block of steel with letter No. 7 after the steel had been annealed to eliminate any possibility of variation in the steel. A burr can also be seen in the bottom of the matrix which would be removed by retracing during the finish cut. Except for the burr and the narrow face, letter B, Fig. 9 is engraved as well as if it were finish cut. With an engraving cutter that would produce the right width of face, there is no doubt that the matrix would finish satisfactorily. Such results are encouraging to the engraver.

With a Bierbaum microcharacter, cuts were made on the three steels; hard, soft and annealed, to determine how the fine cut would make its way across the spheroidized steel rather than to find the microhardness. The cut through the annealed steel is much straighter

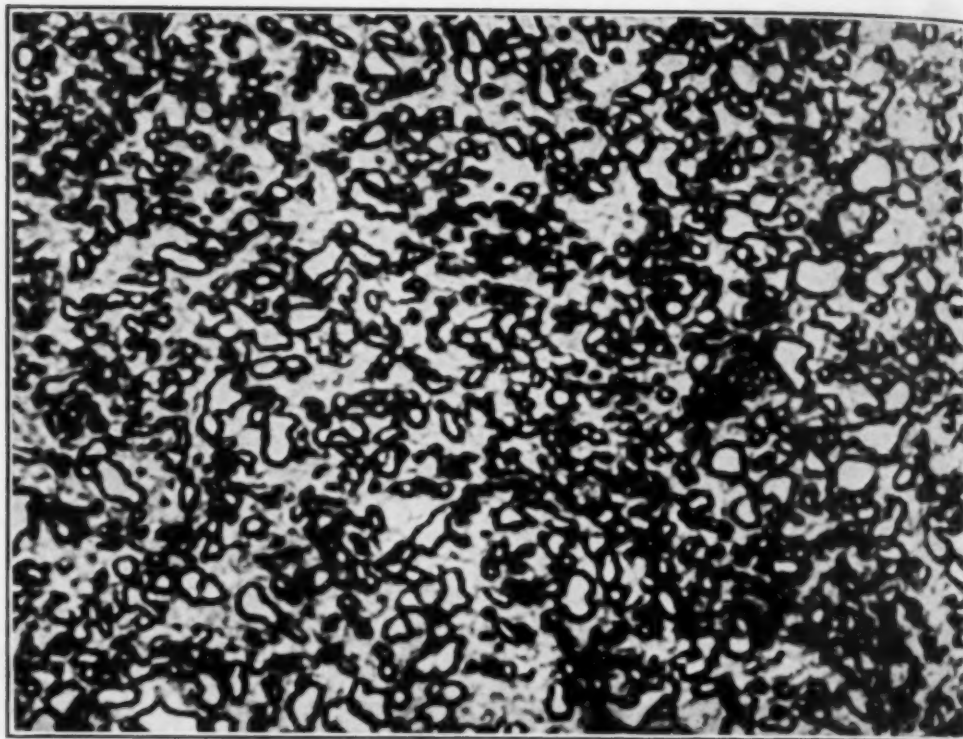


Fig. 13—Photomicrographic Enlargement of Fig. 1, 1500 Diameters, Showing 68 Spheroids Per Square Inch.

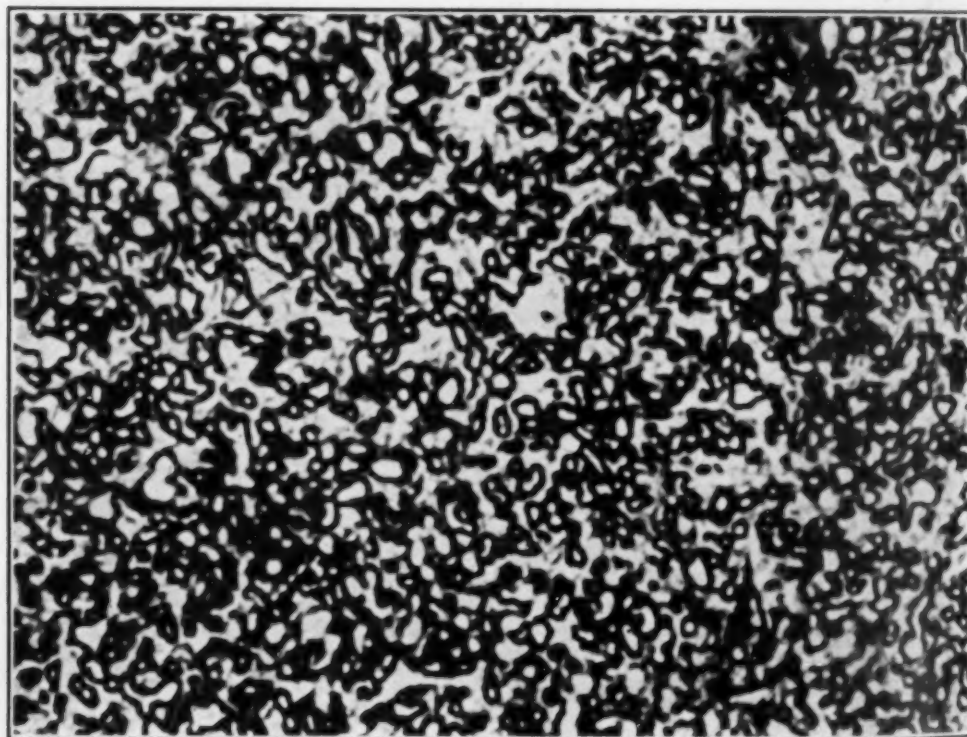


Fig. 14—Photomicrographic Enlargement of Fig. 2, 1500 Diameters, Showing 77 Spheroids Per Square Inch.

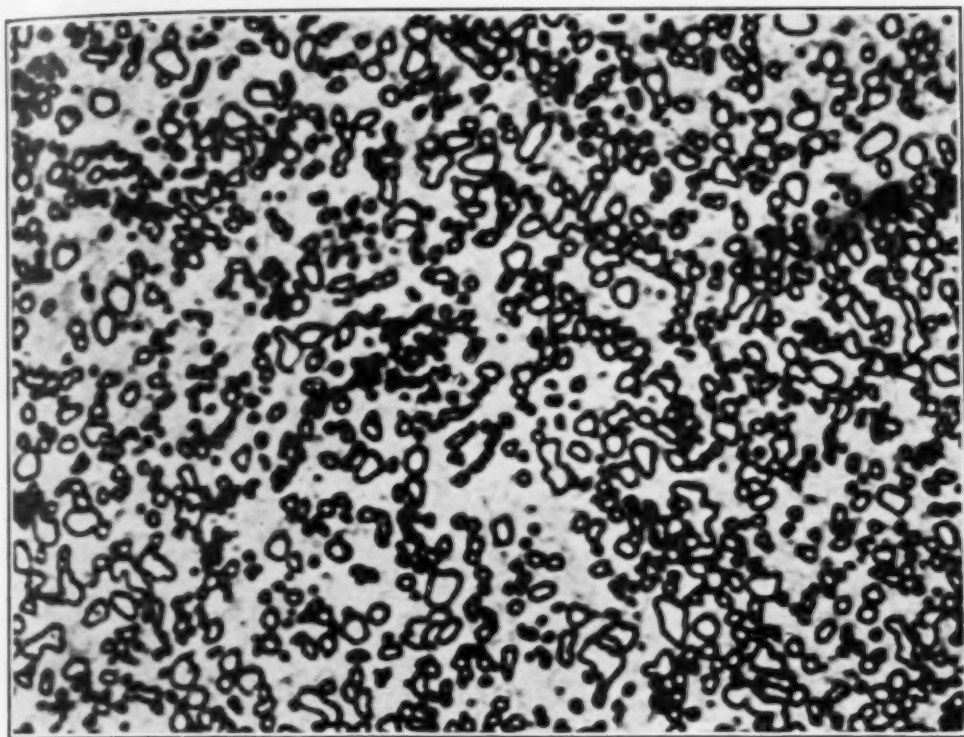


Fig. 15—Photomicrographic Enlargement of Fig. 3, 1500 Diameters, Showing 93 Spheroids Per Square Inch.

than the cuts that slightly wave back and forth across the hard and soft steels. In all three pictures (Figs. 10 to 12), small zigzags appear where the diamond on the end of a cantilever spring has gone around a carbide particle or particles. At the bottom of the cut across the soft steel, the diamond met a spheroid that was either too large or the wrong shape to go around, so it cut its way over the top leaving a cut about  $\frac{1}{4}$  the width of the rest in the mixture of iron and iron carbide, showing how hard a carbide particle is and how well it is locked in the steel. The microhardness of cementite has been determined as 5600 while the hardest carburized steel surface is only 3000. Subjected to a sudden shock indicated by the abrupt change in the width of the microcut the carbide particle shows no indication of displacement. Also, the carbides around which the diamond traveled have held their positions. The diamond must have completely cut out or thrown up into the burr, the carbides that are smaller in diameter than the width of the cut, for the diamond traversed short distances without any waving or variation in the width of the cut and these portions of the steel like the rest were filled with small spheroids. Thus the microcuts show how hard the carbide particles



are and how, due to their greater resistance to being displaced than to being cut, a cluster of large cementite particles would present a microscopic obstacle in the steel that would not show in an ordinary hardness determination. These spheroids would be large enough and hard enough to fracture the cutting edges of extremely sharp or fine cutters.

### CONCLUSION

In conclusion, the three examples which were used to illustrate that large spheroidized carbides, which often occur in groups, cause failure of sharp cutting edges and thus rough machined surfaces, will be summed up.

(1) The hard machining steel was found to have larger spheroidized cementite particles than the tool steel that machined easily.

(2) By breaking down the large spheroids in the hard machining lot of steel with the simple annealing operation of heating for three hours at 1250 degrees Fahr. and cooling in the furnace, the steel was made easy to machine.

(3) The microcharacter was used to explain how it was possible for the large iron carbide particles to fracture the edge of the cutting tool.

### ACKNOWLEDGMENTS

The author appreciates the assistance of Prof. William J. Conley and the use of the University of Rochester's metallographic equipment and Bierbaum microcharacter; also, information concerning the breaking down of spheroidized carbides in plain carbon steel volunteered by Edwin C. Yaw, a graduate student at the University.

A complete discussion of the microcharacter may be found in Volume XXIV, Number 3, TRANSACTIONS of the American Society for Metals, 1936, in the article entitled "The Microcharacter as a Research Tool" by William Conley, Weld Conley, H. King and L. Unger. The microhardness of carburized steel is stated as determined by Bierbaum and of cementite as determined at the University of Rochester laboratory.

### DISCUSSION

**Written Discussion:** By W. H. Wills, metallurgist, Allegheny Ludlum Steel Corp., Dunkirk, N. Y.

The author has presented an interesting description of a special machining operation. The manganese oil hardening die steel referred to is used in a

wide range of sizes from small drill rod up to 8 inches round or more and flats up to 10 or 12 inches in width.

In order to obtain a completely spheroidal structure after hot working, it is the usual practice to normalize at about 1550 degrees Fahr. and anneal at 1375 to 1400 degrees Fahr. followed by slow cooling in the furnace. The Brinell hardness averages 183 to 196. It is more difficult to spheroidize than 1.00 per cent carbon tool steel. If annealed at too high a temperature or cooled too rapidly more or less lamellar pearlite may be present.

When fully spheroidized, the steel machines readily in ordinary tool room operations and even may be handled on automatics. When machining difficulty is encountered, it is usually due to the presence of an excessive amount of lamellar pearlite.

The author did not mention the size of the stock used in connection with the test, also whether any difference was noticed in taking cuts across or parallel with the grain of the stock.

This type of steel is somewhat subject to carbide segregation and when a polished longitudinal section is examined microscopically there is usually some tendency toward banding. Carbide-rich areas with a relatively larger number of spheroidal particles will alternate with those with a less number in the ferrite matrix. The carbide-rich areas usually contain the spheroids of larger size.

As might be expected, the average size of the spheroidal carbides is larger with increasing size of section, although in general the size is somewhat finer than those of carbon tool steel in similar sizes.

In order to check the results obtained by the author with the anneal at 1250 degrees Fahr. samples of the grade ranging in size from  $\frac{5}{8}$  to  $3\frac{3}{4}$  inches round were examined for structure and hardness after the regular anneal. Separate pieces were then reannealed for 3 hours at 1250, 1350 and 1450 degrees Fahr. cooling in the furnace.

Examination for structure and hardness showed that after the 1250 degrees Fahr. anneal, the most uniform distribution of carbides was obtained together with low hardness in line with the author's determinations. At 1350 degrees Fahr. there was more tendency for the carbides to be arranged in groups while after the 1450 degrees Fahr. treatment there was 30 to 40 per cent lamellar pearlite present and a marked increase in hardness.

These results would indicate that around 1250 degrees Fahr. there is apparently a carbide diffusion range while at slightly higher temperature—1350 degrees Fahr.—there is a tendency for the carbides to agglomerate.

### Oral Discussion

W. H. WILLS:<sup>1</sup> I regret that there was not a little more time to go into this further, because I was wondering whether if the 1250 degrees Fahr. anneal was continued longer than three hours, just what the effect would be, whether there was a limit as to the time diffusion would take place and what was the range above or below 1250 degrees Fahr. that this diffusion takes place.

<sup>1</sup>Metallurgist, Allegheny Ludlum Steel Corp., Dunkirk, N. Y.

**Author's Reply**

Mr. Wills has added several valuable statements which greatly increase the scope of the paper and noted a few important omissions that should have been included. Although the section used in the annealing operation was only a half inch thick, the die blocks are cut from a one and a half inch by two inch bar of steel. All of the letters are machined in faces of the steel parallel with the grain and no tests were made on faces across the grain. No indication of banding was found, as may be seen in the photomicrographs, in the paper, which are all parallel with the grain of the stock. A sample of this steel annealed at 1350 degrees Fahr. for three hours showed large irregular bands of carbide in the direction of rolling, bordering pearlite areas from too rapid cooling and iron bands depleted in carbide. I would like to add, in the event that practical use is made of the special annealing process stated in this paper, that the process is reversible; that is, the first three hours heating and cooling breaks down the carbide; the second brings them back to their original size; the third breaks them down again, etc. A continuous six hour heating at 1250 degrees Fahr. evidently decreases and increases the carbide size so that it looks as if it had not been heated. Continuous heat for longer than six hours seems to maintain growth of the carbide; also that 1250 degrees Fahr. is the best temperature to break down the carbide. Professor Conley and I have tested both higher and lower temperatures with varying time intervals. This subject should, however, be covered in a paper by itself and include the hardness values and hardened or normalized steels along with the structures.



## RECOVERY OF COLD-WORKED NICKEL ON ANNEALING

BY ERICH FETZ

### *Abstract*

*The object of this paper was to study further the various factors which may be responsible for the unusually wide temperature range of softening of cold-worked nickel as established by different investigators. The temperature range of hardness recovery from cold working now extends from less than 300 to over 800 degrees Cent.*

*This paper concerns itself with the effects of chemical analysis, origin of testing material, time of annealing, annealing atmosphere, thickness before rolling, and speed of plastic deformation. The effect of these factors on the recovery of nickel was studied by means of hardness measurements.*

IT has been shown in a previous installment (2)<sup>1</sup> on the subject that severely cold-worked nickel of high purity may recover from cold work in the temperature range of 300-400 degrees Cent. instead of 400-500 degrees Cent. as found by Ransley and Smithells (3) and the author (1). Carbonyl nickel was utilized in these experiments, which indicated a pronounced effect of pretreatment on the location and extension of the temperature range of hardness recovery. (See Fig. 5, curve a, b, c, d).

In view of the profound effect of contaminations on the recovery of pure metals from cold work, the synthetic way of preparing solid samples from highly pure powder appears to be very promising. This method circumvents the possibility of contaminating the pure metals during melting and permits studying the effect of individual elements added without disturbing the balance of other impurities present which in their total amount may affect the recovery from cold work. The uncertainties of artificially alloying elements to highly pure metals by the conventional melting process becomes evident by critically examining the pioneer work of Ransley and Smithells (3). The British investigators' addition of 0.07 per cent magnesium to their

<sup>1</sup>The figures appearing in parentheses refer to the bibliography appended to this paper.

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. The author, Erich Fetz, is research metallurgist, Wilbur B. Driver Co., Newark, New Jersey. Manuscript received June 25, 1937.

refined nickel (99.62 per cent) resulted in a shift of recovery temperature from 410 to 620 degrees Cent. Yet commercially pure nickel (98.94 per cent) containing 0.09 per cent magnesium in addition to considerable quantities of further contaminating elements recovers at a lower temperature. ( $T_r = 600$  degrees Cent.). Although the drastic effect of traces of magnesium on the softening of cold-worked nickel has been proved qualitatively, the shortcomings inherent in the experimental technique seem to make quantitative determinations rather difficult.

In the following, a new approach to the problem of the effect of impurities on the recovery of highly pure nickel has been made by intimately mixing the powders, sintering, plastically deforming and long-time annealing. As the contaminating elements, silicon, tin and copper were selected. Ransley and Smithells had previously studied the effect of magnesium, iron and manganese. The quantities employed and the resulting shift of the recovery temperature from that of their refined nickel ( $T_r = 410$  degrees Cent.) is as follows:

0.07 per cent Magnesium	+ 210 degrees Cent.
2.33 per cent Iron	+ 140 degrees Cent.
0.34 per cent Manganese	+ 140 degrees Cent.

#### EFFECT OF SILICON, TIN AND COPPER ON THE RECOVERY OF NICKEL MADE FROM CARBONYL NICKEL POWDER

All powders used were finer than 325 mesh. They were mechanically intimately mixed and hydraulically compressed at 50 tons per square inch in a tool steel die of 1-inch inside diameter. Each slug was sintered individually in a quartz tube placed into a small coil of a high frequency induction furnace. The temperature was maintained for 5 minutes at each of the successive temperatures of 600, 1100 and 1400 degrees Cent. A hydrogen atmosphere was maintained excepting that the tube was evacuated 5 times during the treatment. The object of this method of sintering was to eliminate as much carbon and oxygen from the carbonyl nickel as possible. In order to avoid contamination, the samples rested on the same material.

During sintering, the samples had shrunk considerably. The further treatments and tests performed on the material as it was converted into solid nickel are summarized in Table I. As can be seen, 0.25 per cent and 0.75 per cent silicon, 0.25 per cent tin and 0.75 per cent copper were added to the carbonyl nickel powder. A sample

Table I  
Treatment of Carbonyl Nickel Samples

Sample .....	1	2	3	6
Addition .....	0.25% Si	0.75% Si	0.25% Sn	0.75% Cu
First reduction in rolling, per cent .....	31.8	31.7	31.7	28.3
Hardness after rolling in Brinell units .....	151	156	179	161
Hardness variations in Brinell units .....	147-156	132-169	153-195	145-169
Mean hardness after annealing 100 hrs. at 1000° C. in Brinell units .....	60	65	74	58
Spec. gravity after annealing 100 hrs. at 1000° C. in g/cm <sup>3</sup> .....	8.59	8.174	8.60	8.394
Second reduction (to 0.18 in.) in per cent ..	31.8	32.8	32.8	32.8
Mean hardness after second rolling in Brinell units .....	150	176	159	150
Hardness variations in Brinell units .....	137-159	162-185	147-169	139-156
Hardness after annealing 100 hrs. at 1020° C. in Brinell units .....	64	63	73	60
Spec. gravity after annealing 100 hrs. at 1020° C. in g/cm <sup>3</sup> .....	8.73	8.599	8.6**	8.706
25 per cent .....	145	161	167	145
50 per cent .....	182	184	202	179
75 per cent .....	207	220	225	208
90 per cent .....	216	218	232	214
Recovery temperature in degrees Cent. .... 10 per cent*	559	593	670	568
90 per cent .....	455	487	572	400
Shift of recovery temperature in degrees Cent. by every 10 per cent reduction .....	13.0	13.3	13.5	21.0

\*Extrapolated from Figs. 1-4.

\*\*Specific gravity in relation to further rolling: 25% 8.798  
50% 8.822  
75% 8.860  
90% 8.877

containing 0.75 per cent tin cracked up in the first pass on rolling after sintering. A specimen containing 0.25 per cent copper had to be rejected because overheating in sintering had caused deep edge cracks.

The samples were very cautiously cold-rolled about 32 per cent from 0.387 to 0.264-inch thickness in an experimental rolling mill (rolls  $3\frac{3}{16}$ -inch diameter, speed 12 r.p.m., reductions: 0.005 inch per pass). Hardness measurements showed softness at the periphery. A diffusion anneal of 100 hours at 1000 degrees Cent. was applied. The hydrogen atmosphere in the furnace was completely evacuated twice daily and renewed. The density determinations after this treatment indicate a certain degree of porosity. (See Table I). The density was markedly increased after a second cold rolling of about 33 per cent and diffusion anneal of 100 hours at 1020 degrees Cent. Further density determinations were made on the sample containing 0.25 per cent tin whose relatively low density increased from 8.6 to 8.798, 8.822, 8.860 and 8.877 grams per cubic centimeter respectively due to a reduction in rolling of 25, 50, 75 and 90 per cent. The double recrystallizations and intermediate plastic deformations



had converted the specimens into rather homogeneous and ductile metal so that they could be rolled 25-90 per cent without edge cracking.

The initial thickness of all specimens prior to the final reduction of 25, 50, 75 and 90 per cent was uniformly 0.180 inch. Small samples were cut from the variously deformed specimens and annealed for 30 minutes at different temperatures in a muffle furnace. The samples rested on a copper block of considerable heat capacity providing a uniform temperature and rapid heating to temperature. The temperature was measured with a calibrated platinum, platinum-rhodium thermocouple. The electric furnace was controlled by an unprotected thermocouple built of thin chromel-alumel wires and placed in the vicinity of the resistance winding. Thus the temperature variations in the copper block became negligible.

The hardness was determined with the standard Rockwell machine. On thin samples, the Rockwell Superficial Hardness Tester (RS15T, RS30T, RS15N, RS30N) was employed. Three to ten hardness determinations were made on each specimen. The mean value for each was converted from the respective Rockwell scale to a Brinell hardness by means of a conversion table furnished with the Rockwell instrument. A common basis of comparison was thus obtained.

Whereas the hardness of the hard-rolled and completely annealed samples was quite uniform, a great number of tests were required for arriving at a reliable mean hardness value of samples just passing through the temperature range of pronounced softening. This holds in particular for all specimens of the smallest reduction studied; i.e., 25 per cent. Very frequently, considerable deviations in hardness were found between the top and bottom surfaces of these samples. Both sides of each sample were tested throughout this study.

The effect of rolling and annealing of the compressed powder samples is shown in Figs. 1 to 4. The 3 sections of each figure show respectively; a, the relation of strain hardening to the amount of plastic deformation; b, the relation of the recovery of hardness from cold working to annealing temperature and to amount of reduction in thickness; and c, the softening temperature values derived from the individual softening curves of section b. As the characteristic softening values, those temperatures have been taken at which a sample has lost half of its maximum hardness. This maximum hardness may be the result of cold working or due to the slight hardness

increase preceding the softening of impure grades of nickel (1). It appears that the hardness increase is absent in very pure nickel.

### EFFECT OF SILICON

It is quite obvious that the effect of additions on the softening of pure metals can only be studied in those cases where the additional element remains in solid solution at all times. This point cannot be

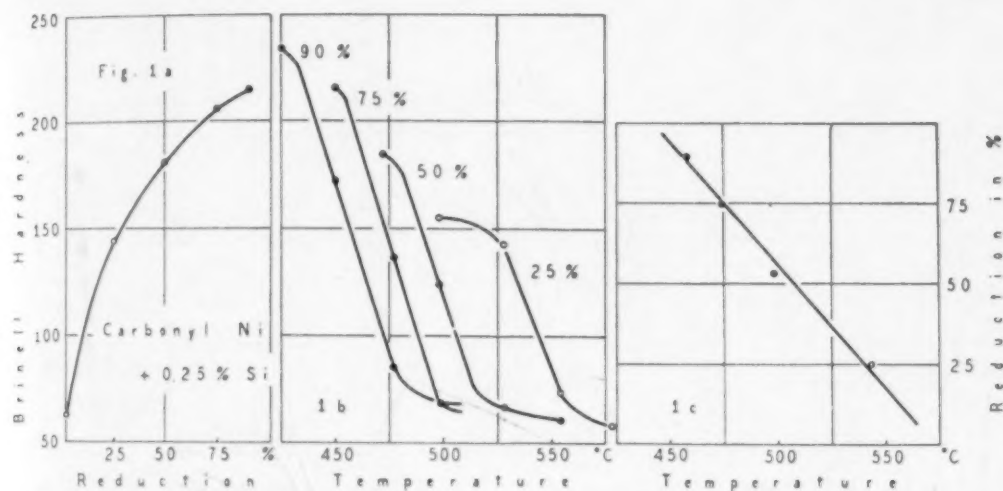


Fig. 1—Recovery of Carbonyl Nickel Containing 0.25 Per Cent Si (30-Min. Anneal).

overemphasized in view of the fact that considerable changes of solubility with rising temperatures have been recently found in cold-worked binary alloys. For instance, contrary to the findings of a score of previous investigators who examined annealed (metastable)

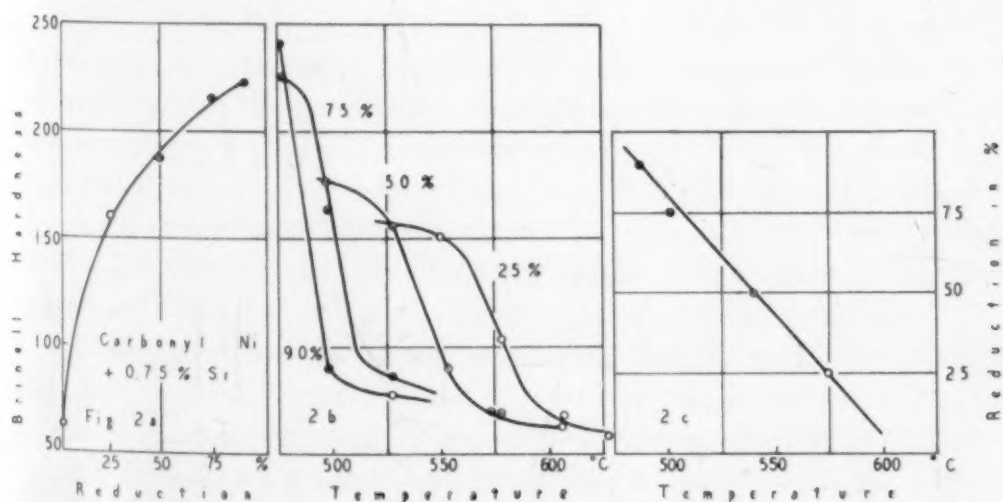


Fig. 2—Recovery of Carbonyl Nickel Containing 0.75 Per Cent Si (30-Min. Anneal).

material, the  $\alpha$ -phase boundary line in the copper-tin and copper-zinc systems has been found to change rapidly with temperature if cold-worked material is employed. (4), (5).

Regarding silicon, it can be said that the maximum addition of 0.75 per cent applied (Fig. 2) remains in solid solution (6). The effect of 0.25 per cent silicon on the recovery is quite pronounced. According to Fig. 1b and c, the softening range of material cold-rolled between 10 and 90 per cent extends from 455 to 559 degrees

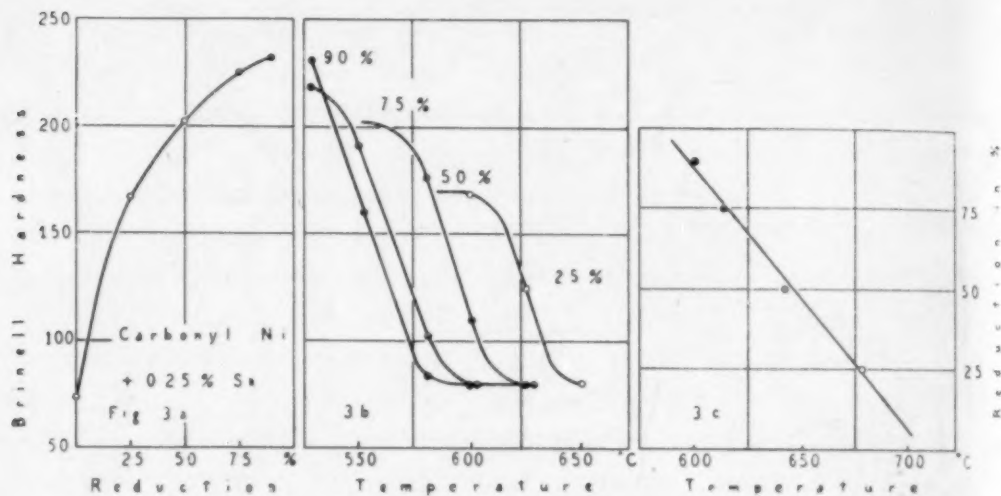


Fig. 3—Recovery of Carbonyl Nickel Containing 0.25 Per Cent Sn (30-Min. Anneal).

Cent. Each increase of reduction by 10 per cent lowers the softening point linearly by 13.0 degrees Cent. Carbonyl nickel samples containing no additions after similar pretreatments recover at temperatures at least 100 degrees lower than sample 1 containing only 0.25 per cent silicon.

Increasing the silicon addition to 0.75 per cent further shifts the temperature range of hardness recovery by about 40 degrees Cent. according to Fig. 2. A sample cold-rolled 10 and 90 per cent recovers at 593 and 487 degrees Cent. respectively. The previously established law (1), (2) of a linear relation between hardness recovery and amount of reduction also holds for this sample. The slope of the  $T_r$ /per cent reduction curve shows that every change of 10 per cent reduction shifts the softening point by 13.3 degrees Cent.

It is quite noteworthy that 0.25 per cent silicon is relatively effective as it shifts the softening points by approximately 100 degrees Cent. (Fig. 5, curve f) while thrice that amount of silicon produces a shift of only 140 degrees Cent. (Fig. 5, curve g).



## EFFECT OF TIN

Contrary to previous findings of Guillet and Voss, recent X-ray determinations disclosed a considerable change of solubility of tin in nickel (7), (8). There seems to be some doubt as to the exact course of the  $\alpha$ -phase boundary line at low (and high) temperatures (9). Nevertheless, on the basis of X-ray pictures taken on severely worked samples after 14,000 hours annealing at 450 degrees Cent., it

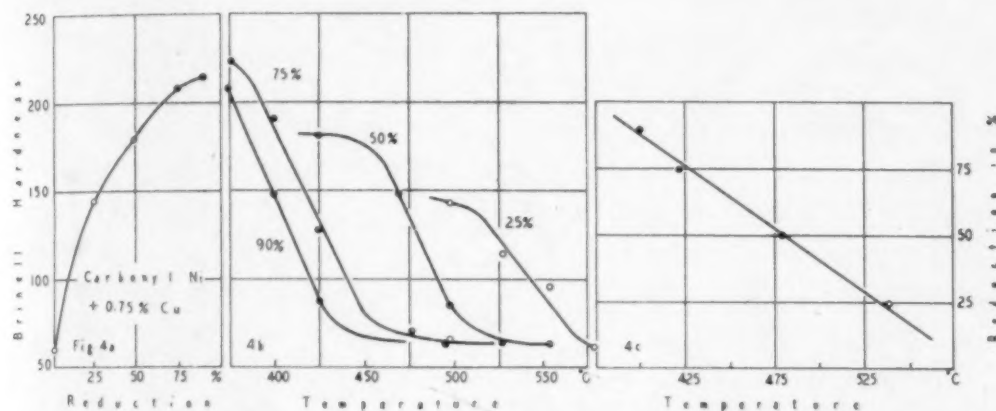


Fig. 4—Recovery of Carbonyl Nickel Containing 0.75 Per Cent Cu (30-Min. Anneal).

can be definitely said that a nickel-tin alloy containing only 0.25 per cent tin remains homogeneous at all temperatures.

Fig. 3a indicates that the addition of 0.25 per cent tin causes a more pronounced strain hardening effect in rolling than the addition of either 0.25 per cent silicon or 0.75 per cent silicon. Figs. 3b and 3c show a rather phenomenal effect of the tin addition: the softening range has been shifted to higher temperatures by 220 degrees Cent. This drastic effect of 0.25 per cent tin in comparison with the relatively mild action of silicon is the more surprising as the atomic weight of tin is 4.23 times greater than the atomic weight of silicon. The reason for the peculiar behavior of tin (and magnesium) cannot be given and many additional recovery tests appear necessary to determine qualitatively and quantitatively which elements act similarly and why.

The hardness recovery-temperature curve (Fig. 3c) is again a straight line and the change of the softening temperatures due to increasing plastic deformations is of approximately the same order (13.5 degrees Cent. per 10 per cent reduction) as was found on other samples made from carbonyl nickel powder.

## EFFECT OF COPPER

The effect of copper on the strain hardening and recovery from cold rolling is shown in Fig. 4a-c. In spite of the relatively large addition of 0.75 per cent copper, the softening point of the 90 per cent cold-rolled sample is shifted by only about 50 degrees Cent. However, in contradistinction to all similarly treated samples, the softening range is much wider. A 10 per cent rolled sample recovers at 568 degrees

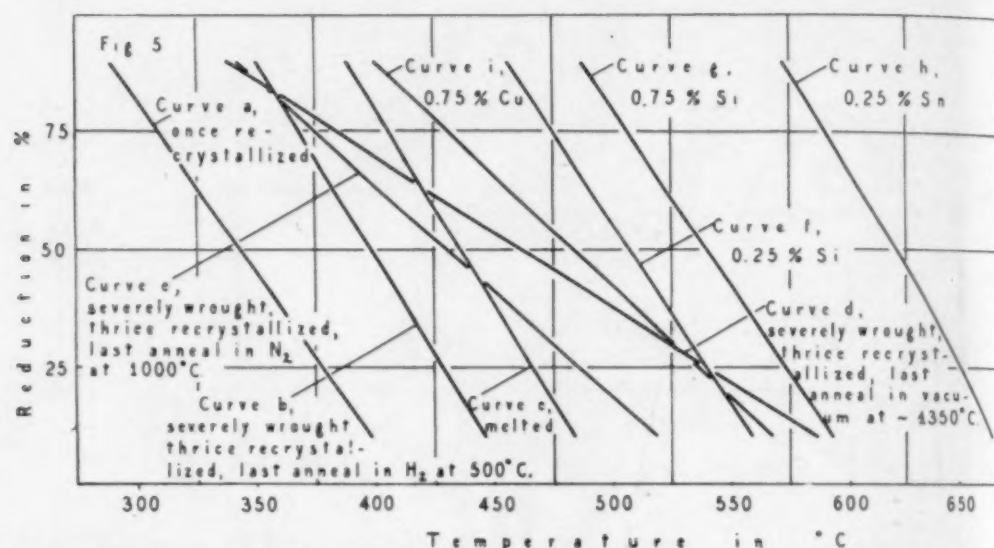


Fig. 5—Recovery Range of Carbonyl Nickel in Relation to Pre-treatment and Alloy Addition (30-Min. Anneal).

Cent. and every change of 10 per cent reduction shifts the recovery temperature by 21.5 degrees Cent. A plausible explanation for this peculiar behavior cannot be offered. Further experiments appear to be necessary before attributing this effect to the addition of copper rather than to the internal structure of the test piece. Samples containing the copper were presumably treated in exactly the same fashion as the samples, the softening characteristics of which are presented in Figs. 1 to 3 and Fig. 5, curves f, g, h.

It may be appropriate to add here that larger additions of copper do shift the softening temperatures of binary nickel-copper alloys. As will be shown in the near future, a 90 per cent cold-worked sample of the Monel metal ratio (70 Ni, 30 Cu) recovers from cold work at 585 degrees Cent. (10).

**Conclusions.** The experiments described prove the feasibility and advisability of studying the effect of elements on the recovery of pure metals by applying the technique of powder metallurgy.

A very pronounced effect of tin on the recovery of nickel was established; 0.25 per cent tin was found to shift the recovery temperatures by more than 200 degrees Cent. Silicon proved to be less effective; 0.25 and 0.75 per cent silicon raise the softening temperature of nickel by about 100 and 140 degrees Cent. respectively. The effect of copper was relatively small. In the severely rolled sample, a shift of the recovery temperature of about 50 degrees Cent. due to the addition of 0.75 per cent copper was observed.

#### EFFECT OF GAS ATMOSPHERE ON THE RECOVERY OF NICKEL

A marked difference in the extension of the softening range was previously found with the hydrogen-treated carbonyl nickel samples on one hand (Fig. 5, curves a, b, c) and the vacuum annealed sample at the other hand, (curve d). This raises the old problem of the rôle of hydrogen in electrolytic nickel and its effect on the energy content of cold-worked metals as proved by injections with radioactive matter (11), (12). In view of the considerable solubility of hydrogen in nickel, it is essential to know whether the widespread use of hydrogen as a protective gas atmosphere is prohibitive in recovery tests. In view of the insolubility of nitrogen in nickel, it appeared to be desirable to repeat the previous tests using an anneal in nitrogen for the final heat treatment before cold rolling. The pre-treatment of this carbonyl nickel powder slug was as follows—compressing, sintering in hydrogen, cold rolling 20 per cent, annealing in flowing hydrogen for 90 hours at 1030 degrees Cent., cold rolling 40 per cent, hydrogen annealing for 100 hours at 1000 degrees Cent., cold rolling 50 per cent to final thickness of 0.18 inch. The sample was cut into three pieces. One section was recrystallized in flowing hydrogen for 5 hours at 500 degrees Cent. The recovery range is shown by curve b, Fig. 5.

An analysis of this sample yielded the following results:

Cobalt	less than 0.001 per cent
Copper	= 0.005 per cent
Iron	= 0.045 per cent
Silicon	= 0.001 per cent.

The carbon determination according to the Yensen method showed 0.002 per cent carbon as occluded gas, obtained by heating in vacuum at 600 degrees Cent. for 10 minutes, and 0.0045 per cent carbon obtained by burning in oxygen at 1150 degrees Cent.



Another section of this carbonyl nickel was annealed for 30 minutes at between 1300 and 1400 degrees Cent. in a vacuum. According to curve d, Fig. 5, the recovery range of this sample is much wider. Only the severely rolled samples of both materials recover at the same low temperatures.

A third section of this material was recrystallized in nitrogen. The sealed furnace containing the sample was heated to 1000 degrees

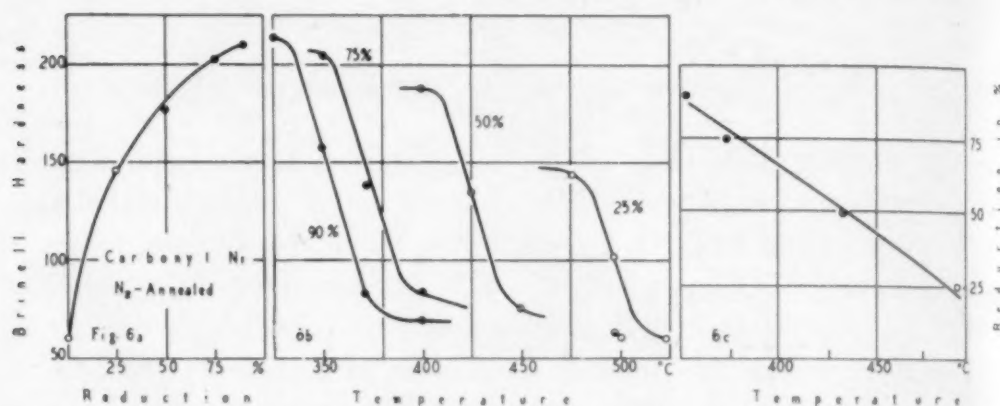


Fig. 6—Recovery of Severely Wrought, Thrice Recrystallized Carbonyl Nickel (Last Anneal in Nitrogen at 1000 degrees Cent.).

Cent. in 2 hours, held at this temperature for 1 hour and cooled to 200 degrees Cent. After this final anneal, the specimen was now cold-rolled in the same fashion as observed in previous tests. The recovery characteristics are graphically presented in Fig. 6. The most conspicuous result is that in contradistinction to the hydrogen-annealed carbonyl nickel powder samples, the softening range is considerably wider although that of the vacuum-treated metal has not been reached. The 90 per cent cold-worked sample recovers at 342 degrees Cent., while a 10 per cent reduced specimen loses half of its hardness at 520 degrees Cent. Thus every increase of reduction by 10 per cent lowers the softening temperature by 21.3 degrees Cent. It should not be overlooked that the recovery temperature range of the 3 differently treated carbonyl nickel samples is the wider the higher the temperature of the last annealing prior to the final cold rolling:

Final Annealing Treatment			Recovery Range of 10-90 Per Cent Rolled Carbonyl Nickel Degrees Cent.	Shift of Tr Temperatures by 10 Per Cent Reduction Degrees Cent.
Atmosphere	Temperature Degrees Cent.	Time		
Hydrogen	500	5 hrs.	350-450	12.7
Nitrogen	1000	1 hr.	342-520	21.3
Vacuum	1300-1400	30 min.	338-586	31.0

It appeared advisable to repeat the experiment with electrolytic nickel. Two grades were used, briefly called A and B. A 4 by 4 inch piece was milled on both sides and cut into strips  $1\frac{1}{4}$  inches wide. The thickness of the samples was 0.265 inch. One sample of each was brought up to 1000 degrees Cent. in 3 hours in a vacuum, in hydrogen, and in nitrogen, held at this temperature for one hour and then furnace-cooled in about 2 hours. The differently treated mate-

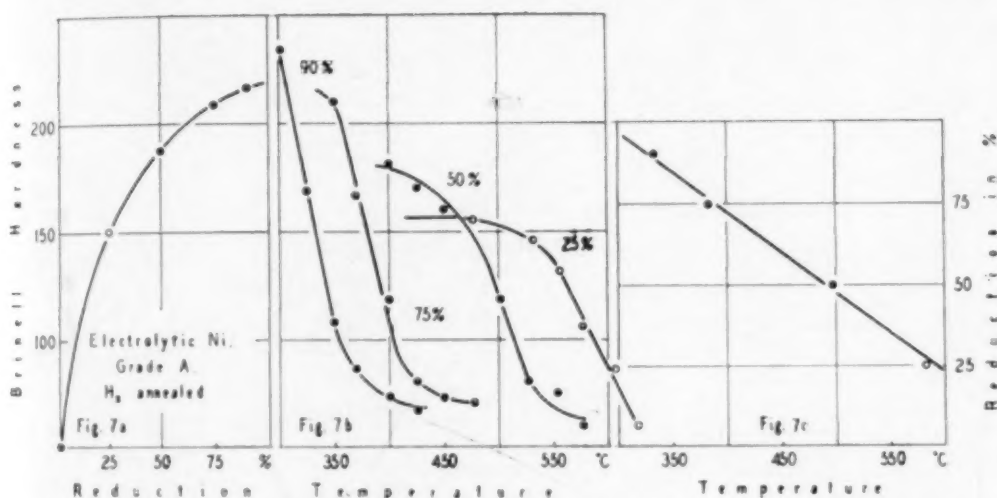


Fig. 7—Recovery of  $H_2$ -Annealed Electrolytic Nickel, Grade A (30-Min. Anneal).

rials were cold-rolled in the same fashion by 25, 50, 75 and 90 per cent and small samples were annealed for 30 minutes at various temperatures. The experimental results with the grade A nickel on the recovery of the hydrogen, vacuum, and nitrogen-treated material are given in Figs. 7, 8 and 9 respectively. The most conspicuous result is that there is practically no difference in the recovery of the variously treated materials. Furthermore, the softening range of these samples is unusually wide, every increase of 10 per cent in reduction of thickness shifting the softening point by 40 degrees Cent. (The scale of the abscissae is half of that in Figs. 1, 2, 3, 4 and 6.) It is furthermore quite noteworthy that this commercial brand of electrolytic nickel softens below 400 degrees Cent. if cold-worked more than 75 per cent.

The second grade of electrolytic nickel, grade B, used in these tests has been previously described (1). The recovery of the material annealed in a nitrogen atmosphere and in a vacuum and cold-worked in the same manner as the grade A described above is shown in Figs. 10a-c and 11a-c respectively. A marked difference may be

Shift of Tr.  
temperatures  
10 Per Cent  
Reduction  
degrees Cent.  
12.7  
21.3  
31.0

noted from these figures. The vacuum-treated material softens over a wide range, while the material annealed in nitrogen softens in a comparatively small temperature range. The vacuum annealed material, incidentally, recovers from cold work in almost exactly the same fashion as has been previously found (1) on this material after 20 per cent reduction and recrystallization in a vacuum. The 90 per cent cold-worked sample recovers at 446 degrees Cent. By extrap-

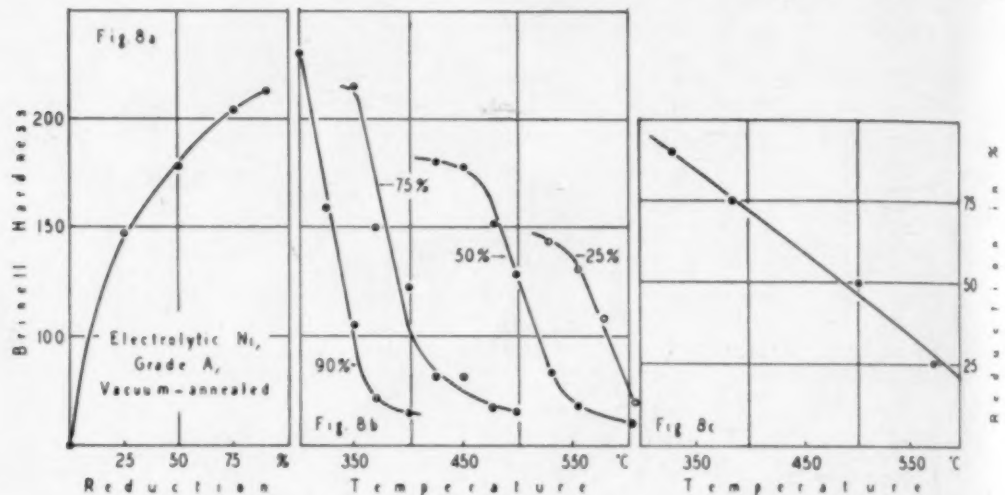


Fig. 8—Recovery of Vacuum-Annealed Electrolytic Nickel, Grade A (30-Min. Anneal).

olation from the curve of Fig. 11c, the softening point of a sample reduced 10 per cent would be 632 degrees Cent. A shift of the recovery temperature of 22.8 degrees Cent. is brought about by every 10 per cent of reduction.

The softening range of the metal annealed in nitrogen extends from 442 to 559 degrees Cent. with reference to a 10-90 per cent reduction. Why every 10 per cent reduction shifts the softening points by only 13.9 degrees Cent. is hard to explain. Both grades of electrolytic nickel A and B have been annealed together in nitrogen and in a vacuum. Since there is no difference between the vacuum and nitrogen annealed samples of electrolytic nickel, grade A, in contradistinction to grade B, it must be assumed that it is the internal structure rather than the effect of the gas atmosphere which brought about the different behavior of the grade B samples annealed in nitrogen and in a vacuum.

#### EFFECT OF INITIAL THICKNESS

In previous experiments with samples made from carbonyl nickel powder, a distinct effect of the initial thickness upon the



recovery was established (2). Two samples cold-rolled to the same extent, but from different initial thicknesses, did not soften in the same fashion. The sample rolled from the greater thickness recovered at a lower temperature. However, the thinner sample had obtained two additional cold working and annealing treatments. Both samples may thus not be comparable structurally. It appeared to be desirable to repeat the experiment on electrolytic nickel with the view

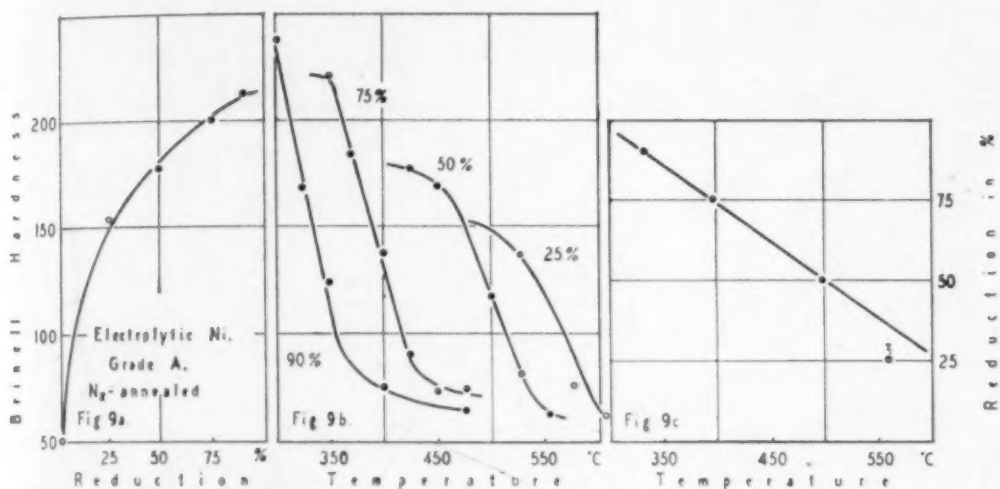


Fig. 9—Recovery of N<sub>2</sub>-Annealed Electrolytic Nickel, Grade A (30-Min. Anneal).

of deciding whether a statement on the per cent reduction represents a sufficiently accurate characterization of the cold work induced by the plastic deformation.

Electrolytic nickel, grade A, was employed for this test. From an initial thickness of 0.267 inch, one specimen was rolled to 0.187 inch or 30 per cent, while the other part was rolled to 0.107 inch or 60 per cent. Both samples were then placed into a furnace running at 1000 degrees Cent. and annealed in flowing hydrogen for one hour. The specimens cooled with the furnace.

Both samples of different thickness were cold-rolled 25 to 90 per cent. The sample reduced 90 per cent from the 0.107-inch specimen had to be discarded as it was impossible to secure reliable hardness measurements on this sample of 0.0106-inch thickness. The recovery of the 25-90 per cent cold-rolled samples derived from the 0.187-inch specimen is presented in Fig. 12a-c and that of the thinner sample in Fig. 13a-c. As can be seen, there is no difference in the recovery for the two samples. With respect to the method of preparation and testing conditions applied in this test, the per cent reduction adequately expresses the amount of cold work induced.

Since this result does not agree with previous tests on remelted carbonyl nickel, the latter experiment was repeated, by making a great number of hardness determinations in the range of pronounced softening and by testing both sides of the samples. The redetermined softening range proved to be much narrower than previously found. (30-minute anneal curve in Fig. 15b.) The recovery points of 383 and 386 degrees Cent. of two samples rolled to the same extent but

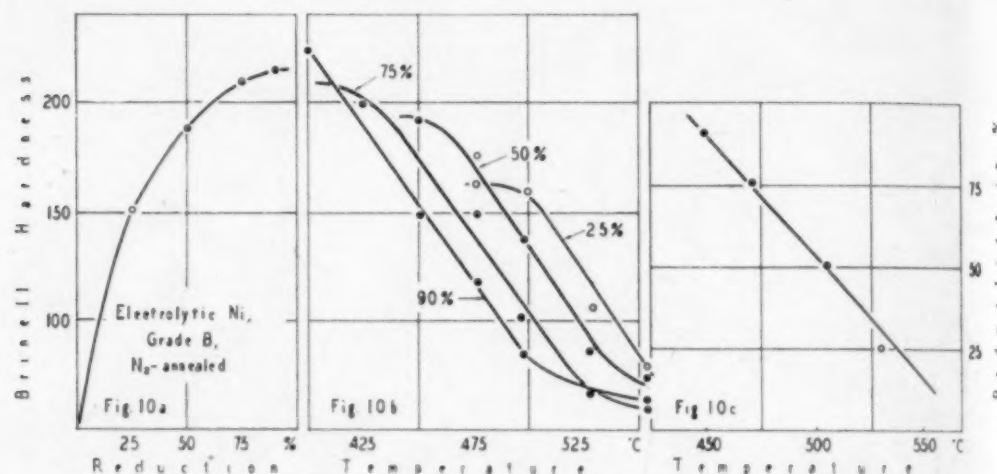


Fig. 10—Recovery of N<sub>2</sub>-Annealed Electrolytic Nickel, Grade B (30-Min. Anneal).

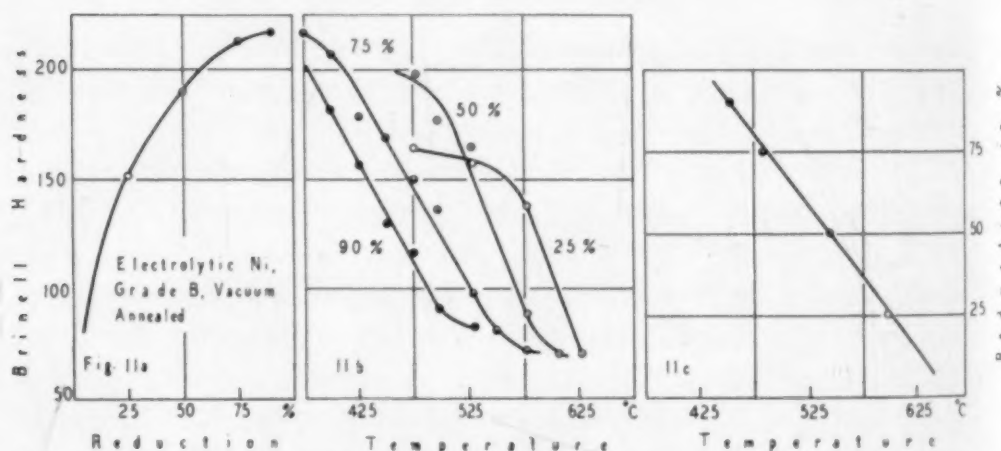


Fig. 11—Recovery of Vacuum-Annealed Electrolytic Nickel, Grade B (30-Min. Anneal).

from different initial thicknesses of 0.294 inch and 0.176 inch respectively are very close together.

**Conclusions.** The thickness prior to cold rolling does not affect the recovery of melted nickel and recrystallized electrolytic nickel cold-rolled to the same percentage. Information on the initial thickness of nickel made from sintered carbonyl powder should be given alongside the per cent reduction as structural differences are apt to occur.

# EFFECT OF DEFORMATION SPEED

In spite of the relatively impure grade of nickel which Schottky and Jungbluth (13) used in their upsetting tests, the recovery temperatures they found are comparatively low. It appears that their method of plastic deformation, in particular the deformation speed, was largely responsible for this behavior.

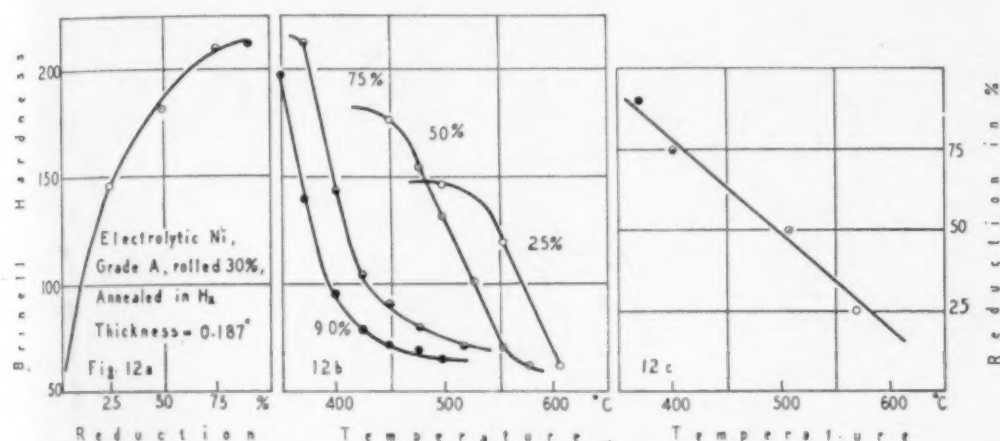


Fig. 12—Recovery of Rolled and Recrystallized Electrolytic Nickel, Grade A, Thickness: 0.187 In. (30-Min. Anneal).

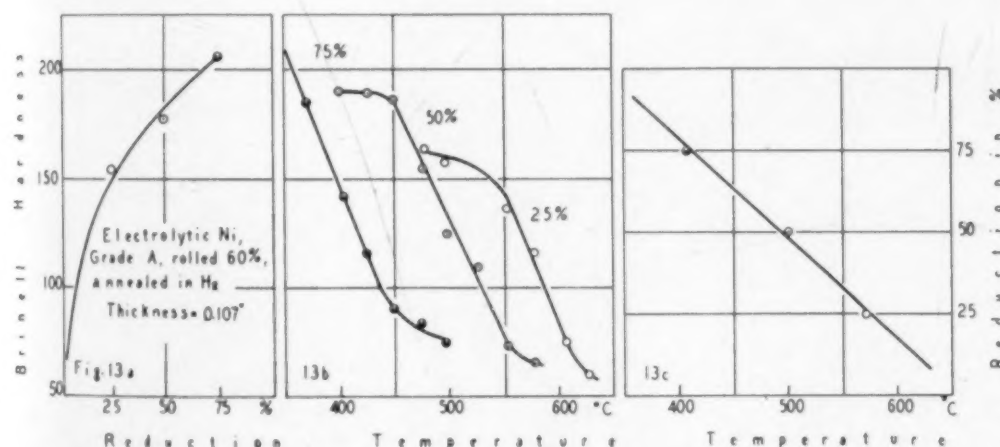


Fig. 13—Recovery of Rolled and Recrystallized Electrolytic Nickel, Grade A, Thickness: 0.107 In. (30-Min. Anneal).

In order to study the effect of deformation speed on the recovery, two samples of the electrolytic nickel, grade B, were cold-rolled about 75 per cent in two passes in a 10-inch, 2-high rolling mill running at 24 revolutions per minute. In Fig. 14, curve "a" shows the softening of the sample annealed in nitrogen and cold-rolled in two passes 79 per cent (first pass was 50.4 per cent). Curve "b" presents the recovery of a vacuum-annealed sample reduced 75 per cent (first



pass 48.6 per cent). The recovery points of the corresponding samples from the same stock which were rolled slowly at 0.005-inch reductions in a small experimental rolling mill (2-high,  $3\frac{3}{16}$ -inch diameter rolls, 12 revolutions per minute) are, according to Figs. 10 and 11, 472 and 452 degrees Cent. for the nitrogen and vacuum-annealed specimen respectively. The faster reduction speed thus lowered the recovery point by 29 and 22 degrees Cent.

*Conclusion.* The effect of deformation speed in rolling on the recovery of nickel appears to be relatively small in comparison with other factors as, for instance, chemical analysis, internal structure and amount of plastic deformation.

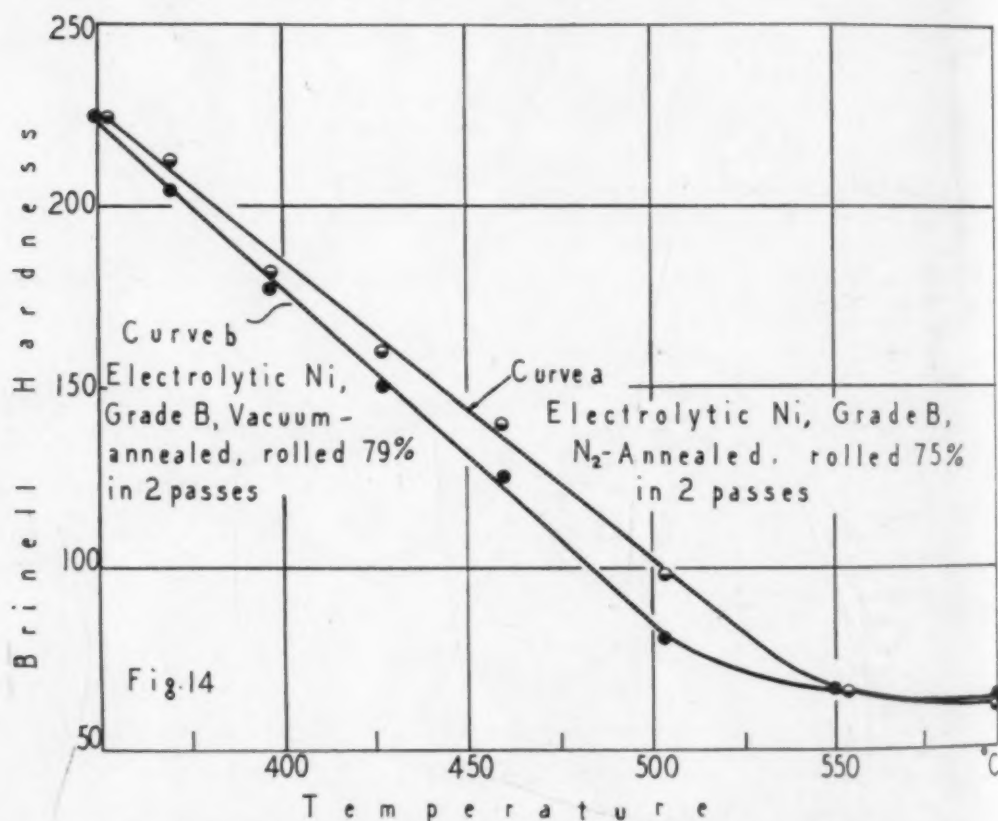


Fig. 14—Recovery of Electrolytic Nickel, Grade B, Rolled at High Deformation Speed (30-Min. Anneal).

#### EFFECT OF TIME

All previous experiments on the cold-worked samples are based on a standard annealing time of 30 minutes duration (1), (2). In the softening tests performed by other investigators on cold-worked nickel, the annealing time varies between 5 minutes and 1000 hours. A study of the effect of time appeared to be desirable. It would be

of interest to know whether any predictions as to the behavior at any temperature could be derived from a series of long-time annealings and correlated to other investigations on the recovery from cold work. It was also the object of the following tests to learn whether there is any difference in the softening of melted metal on

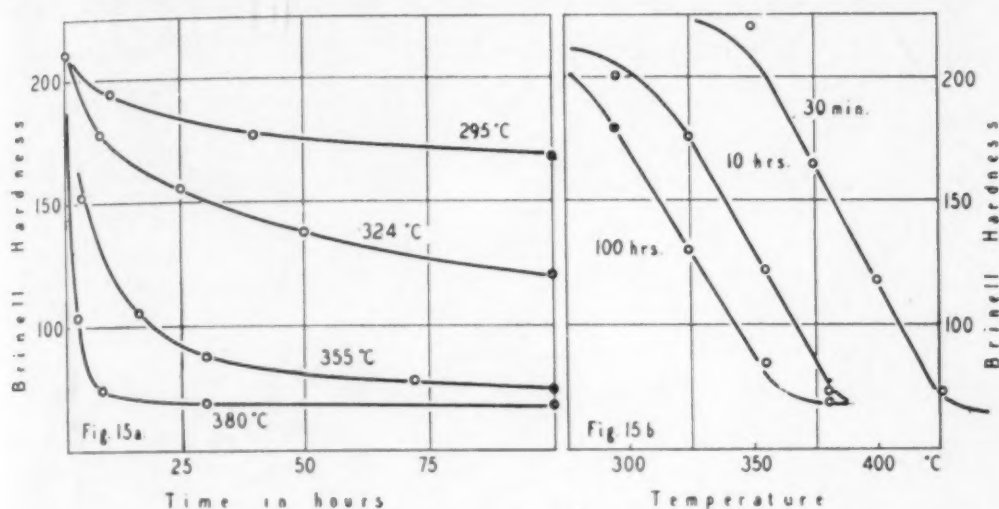


Fig. 15—Softening of Melted Carbonyl Nickel in Relation to Temperature (100-Hr. Anneal).

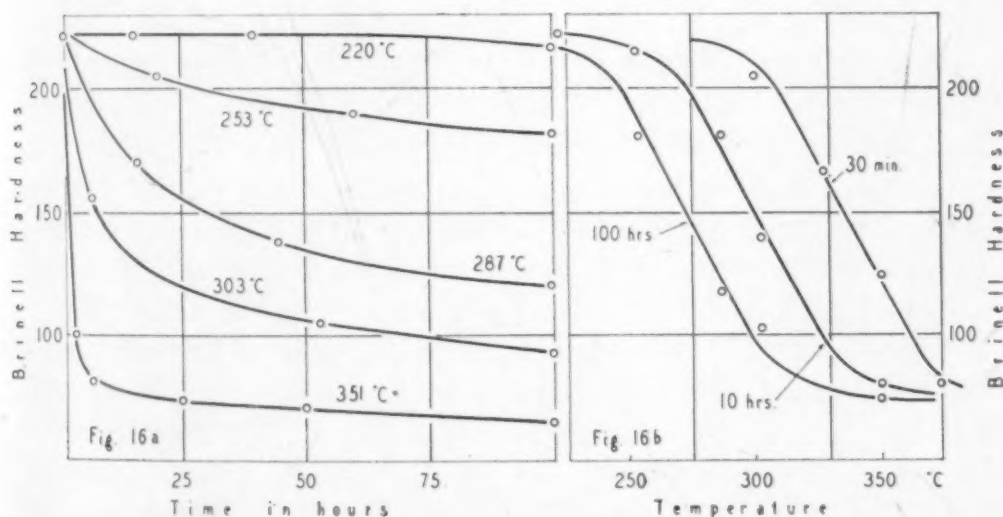


Fig. 16—Softening of Severely Deformed, Thrice Recrystallized, Vacuum-Annealed Carbonyl Nickel in Relation to Temperature (100-Hr. Anneal).

one hand and material made from powder and plastically deformed and recrystallized in a progressive manner on the other. To the best knowledge of the author, such long-time annealing tests have not been published on metallic materials made from powder.

Guillet (14) extended part of his recovery tests (tensile

strength) to 4 hours and failed to find any softening at 550 degrees Cent. Nickel samples cold-worked between 4.4 and 80 per cent were held at 500 degrees Cent. for 1000 hours by Crawford and Worthington (15). The 59 and 80 per cent cold-worked samples had completely softened at the end of this treatment. At the end of a 100-hour treatment, none of from 4 to 80 per cent reduced nickel samples has completely lost its work hardness. Unfortunately, no

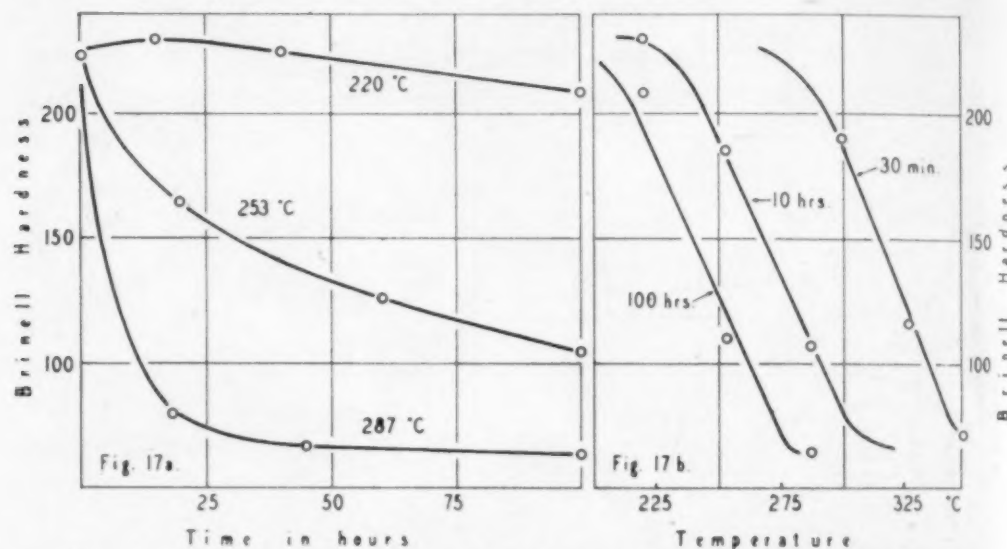


Fig. 17—Softening of Once Recrystallized Carbonyl Nickel (Rolled from 0.60 In. Thickness) in Relation to Temperature During 100-Hr. Anneal.

correlation of the 1000-hour annealing test to their short-time tests is possible. All specimens were first brought up to 578 degrees Cent. On further gradual heating to over 1000 degrees Cent., samples were withdrawn at various "annealing" temperatures. This unusual procedure raises the softening temperatures very materially.

In the following experiments, variously treated carbonyl nickel samples prepared as described below were annealed for 100 hours in a furnace running at a constant temperature. At least three different annealing temperatures were employed for each type of material. At various intervals, the samples were temporarily withdrawn and tested for hardness. The results are shown graphically in Figs. 15 to 18. From the hardness/time curves, given in the a-section of the figure, are derived the hardness/temperature curves of the section b by plotting the hardness values after 10 hours and 100 hours annealing. The recovery curves based on 30-minute annealing have been previously published (2). The characteristic softening values derived from the curves presented in the b-sections are summarily given in



Fig. 19. A 50 per cent loss of work hardening during a 100-hour anneal is shown for the four carbonyl nickel specimens prepared as follows:

The results shown in Fig. 15 are from a sample prepared from carbonyl nickel, pressed, sintered in hydrogen, cut into small pieces, melted under hydrogen in an alundum crucible, the subsequent button cold-rolled 30 per cent, annealed in hydrogen at 1030 degrees Cent.

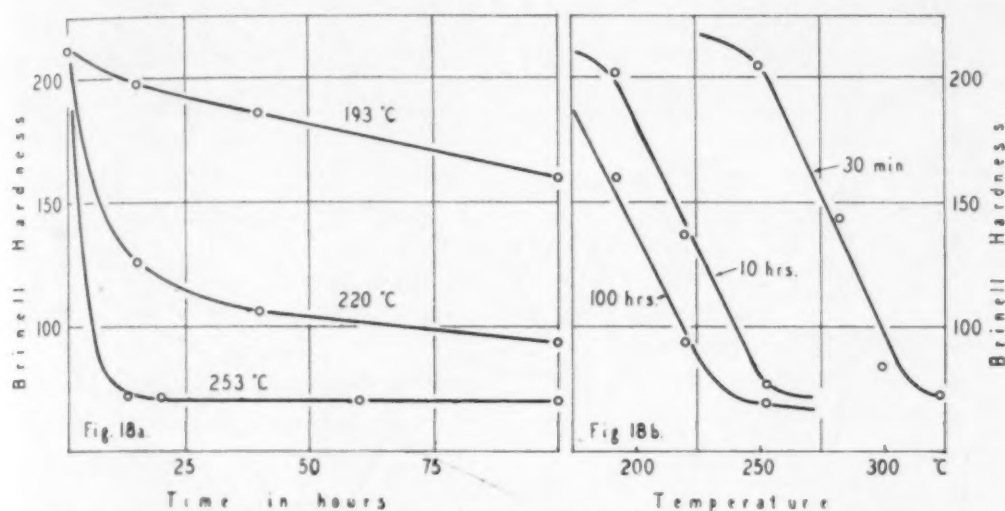


Fig. 18—Softening of Once Recrystallized Carbonyl Nickel (Rolled from 0.173 In.) in Relation to Temperature During 100-Hr. Anneal.

for 90 hours, and finally cold-rolled 94.6 per cent from 0.294-inch thickness. Samples were annealed for 100 hours at 295, 324, 355 and 380 degrees Cent.

It is noteworthy that softening begins below 300 degrees Cent.; this is the lowest softening temperature ever recorded on nickel which has gone through the liquid state. The  $T_r$  values found were 318 degrees Cent. for a sample annealed for 100 hours and 348 degrees Cent. for a sample given a 10-hour anneal. The 30-minute softening curve was redetermined, yielding a  $T_r$  value of 383 degrees (instead of 368 degrees Cent. as previously found)<sup>2</sup>. Nickel from the same stock, but rolled from a smaller initial thickness of 0.1762 inch indicates also a recovery point of 386 degrees for a 94.6 per cent reduction after a 30-minute anneal by extrapolation of curve c, Fig. 5. One additional plastic deformation and recrystallization and the subsequent rolling from a smaller thickness apparently does not change the recovery temperature for a material that has been in the liquid state.

The results presented in Fig. 16 are based on a sample made

from carbonyl nickel, compressed, sintered in hydrogen with several evacuations, rolled 20 per cent longitudinally after machining opposite sides of the slug, and annealed in hydrogen at 1030 degrees Cent. for 30 minutes. The sample was finally rolled 90 per cent from a thickness of 0.18 inch. Hardness measurements were made

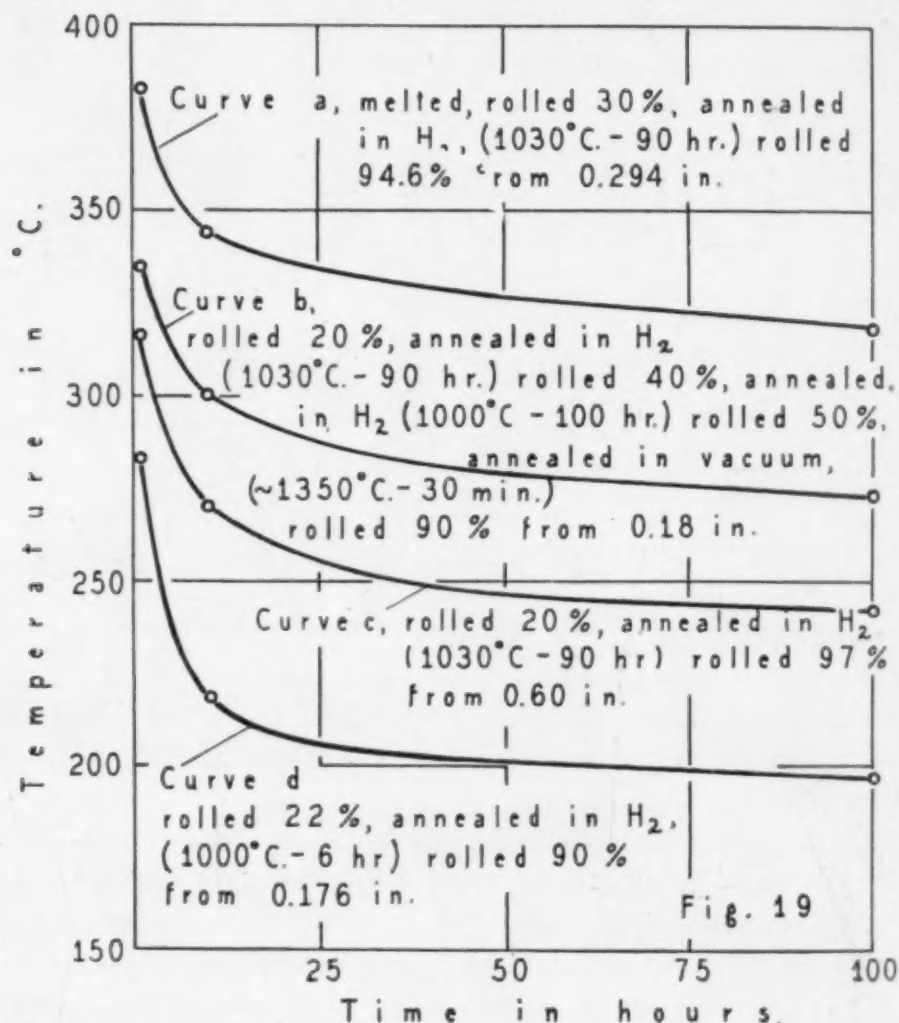


Fig. 19—Loss of 50 Per Cent of Work Hardening of Variously Treated Carbonyl Nickel During 100-Hr. Anneal.

during 100-hour anneals at 220, 253, 287, 303, and 351 degrees Cent. A 50 per cent loss of hardness is effected by a 30-minute anneal at 335 degrees Cent. The same result is obtained at 300 and 273 degrees Cent. respectively during a 10-hour and 100-hour annealing treatment.

In Fig. 17 are shown the results obtained on carbonyl nickel, compressed, sintered in hydrogen with frequent evacuations, planed on opposite sides and rolled longitudinally 20 per cent, annealed in

hydrogen for 90 hours at 1030 degrees Cent., cold-rolled 97 per cent from a thickness of 0.60 inch. Some edge cracking occurred at about 70 per cent reduction of thickness. The annealing temperatures used in the 100-hour heat treatment were 220, 253 and 287 degrees Cent. Extending the annealing time from 30 minutes to 10 hours and 100 hours shifts the softening (Fig. 17b) into the temperature range of 200-300 degrees Cent.

Fig. 18 shows the softening of carbonyl nickel which was pressed, sintered in hydrogen, rolled 21.7 per cent, annealed in hydrogen at 1000 degrees Cent. for 6 hours and then cold-rolled 90 per cent from a thickness of 0.173 inch. The recovery point of this material annealed for 30 minutes was previously found at 283 degrees Cent. It is quite surprising that a 10-hour and a 100-hour annealing shift the recovery temperature to 219 and 197 degrees Cent. respectively.

It can be inferred from Fig. 19 that the tendency toward softening is least pronounced in the melted carbonyl nickel (curve a) and the vacuum treated metal which had undergone the greatest number of plastic deformations and thermal treatments (curve b). The final anneal at 1300-1400 degrees Cent. seems to have thoroughly obliterated the origin of the material. Softening curves c and d of Fig. 19 do not follow the same time law as that of the melted material. The structural differences are probably responsible for this result. The materials represented in curves c and d have apparently been insufficiently wrought and recrystallized to be comparable with solid nickel which has passed through the liquid state. During the initial stages, the stress distribution and the storage of latent energy during plastic deformation in metallic material prepared from powders is apparently different from that of solid metals. The larger internal surface of such pressed and sintered bodies has also been experimentally determined by Werner (11) by means of radio-active measurements. It is interesting that this assumed increase of internal free energy in cold-worked, pressed and sintered shapes made from metal powder does not manifest itself in an increased hardness.

#### TIME-TEMPERATURE LAW OF RECOVERY OF NICKEL

Van Liempt (16), (17) has derived a formula expressing "identical stages of recovery from cold working"

$$T (13.43 + \log t) = C$$



wherein  $T$  = absolute temperature in degrees Cent.,  $t$  = time in seconds and  $C$  is a constant. He has checked his formula with literature data referring to the recrystallization and recovery of thermal electromotive force of copper, recovery of temperature coefficient of electrical resistance of tungsten, softening of cold-worked brass, etc. On the strength of recovery tests on cold-worked nickel, van Liempt "has gained the impression that (his) formula would also hold for this case," but no statement as to what property was studied is given. For attainment of constant X-ray line sharpness of cold-worked nickel, Wilson and Thomassen (18) confirmed van Liempt's relation between time and temperature of annealing.

It appeared desirable to apply the formula given above to the annealing results obtained on the melted carbonyl nickel. A 50 per cent loss of the induced work hardening at 380, 355 and 324 degrees Cent. respectively requires 75 minutes, 16 hours and 41 hours (see Fig. 15). Evaluating the constant  $C$  for these 3 cases yields, 11,150, 11,400, 11,090. The values agree well and it is now possible to determine the time required for any temperature to relieve cold-worked (melted) carbonyl nickel of half of its strain hardening.

Taking a mean value of 11,213 for  $C$ , the melted carbonyl nickel, cold-rolled 94.6 per cent would soften to 141 Brinell at say 295 degrees Cent. in 26 days and 12 hours. It is needless to say that the formula holds for any other state of recovery of the melted nickel, but it seems to be useless to apply van Liempt's formula to the pressed carbonyl nickel results. According to Fig. 19, the time-temperature law of the recovery of these powder samples is obviously different from that of melted material.

#### SOFTENING OF ELECTRO-DEPOSITED NICKEL

If the assumption that the fine grain size in these synthetic metallic bodies in the semi-dense state is responsible for the low recovery temperature, then it should be possible to observe the same phenomenon on electrolytic nickel whose grain structure cannot be often readily resolved under the microscope. Without enlarging on the contested problem whether the abnormal hardness of certain electrolytically deposited metals is due to the hydrogen content or to the ultra-fine grain, the fine grain, which may be accompanied by lattice stresses, has been unequivocally established by X-rays (19). Recent careful experiments (20) disclosed an increase of hardness with diminution in grain size.

The softening curve of the electrolytic nickel, grade A, is shown in Fig. 20 which also presents the softening curves of electrolytic nickel as recently determined by French experimenters (21), (22). There are apparently some differences in the softening characteristics. The loss of 50 per cent of the as-deposited hardness is reached at 475 (Guichard and co-workers), 496 (Jenicek) and 508 degrees Cent. (Fetz).

Samples of electrolytic nickel, grade A, were cold-rolled 75

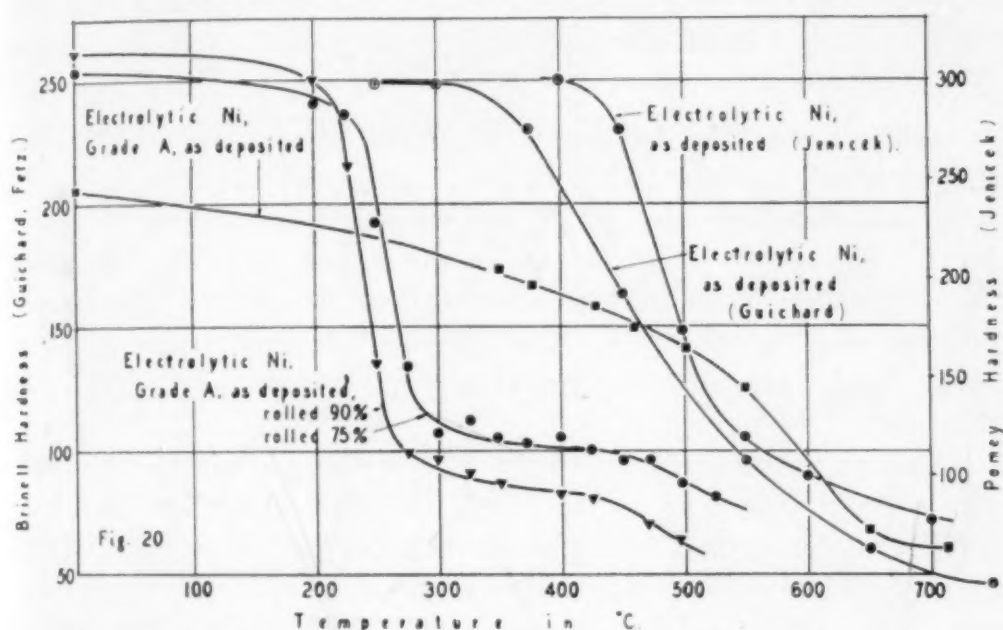


Fig. 20—Softening of 90 Per Cent and 75 Per Cent Cold-Rolled Electrolytic Nickel, Grade A, and Softening of 3 Brands of Electrolytic Nickel.

and 90 per cent from a thickness of 0.265 inch. This was done without any difficulties. The softening curves are given in Fig. 20. Pronounced softening begins at about 200 degrees Cent. The recovery curves flatten out again around 300 degrees Cent. Between 325 and 475 degrees Cent. the hardness does not change materially. The softness of the fully annealed state has not been attained yet. At 475 and 500 degrees Cent., the hardness of the 90 and 75 per cent cold-worked sample drops again notably.

The most conspicuous result of this experiment is that the softening point of the as-deposited electrolytic nickel, grade A, is lowered from 508 degrees Cent. to 255 and 238 degrees Cent. due to a plastic deformation of 75 and 90 per cent respectively. In contradiction to Jenicek's findings (22) on nickel in the electrodeposited state without cold working, the electrolytic nickel sample cold-rolled 90

per cent (Fig. 20) begins to recrystallize at the same low temperature (228 degrees Cent.) at which pronounced softening starts (23).

It is very doubtful that a change of chemical analysis has taken place during recrystallization of this material in vacuum (Fig. 8), in hydrogen (Fig. 7) or in nitrogen (Fig. 9). Therefore, the unusually low recovery points of the electrolytic nickel cold-rolled as deposited, illustrate well the significance of the internal structure (grain size) of the tested material upon the recovery from cold working!

### DISCUSSION

On all pure and contaminated grades of nickel studied so far, the previously established law (1) was confirmed that there is a direct relation between the amount of cold work and the recovery temperatures. The recovery temperature of the 90 per cent cold-rolled samples is usually slightly off the curve. Due to the extreme thinness and the resulting "anvil effect," the specimens appear to be somewhat harder and their softening curve is slightly shifted to higher temperatures.

Although the extension of the recovery range was found to vary substantially depending on the origin and pretreatment of the nickel, it could not be verified in any of the 17 cases studied so far, that "the hardness recovery of nickel depends but little on the amount of cold work" as postulated by Tammann and Moritz (24). The smallest shift of the recovery temperature was found on melted carbonyl nickel; namely 12.3 degrees Cent. per every 10 per cent of reduction. In electrolytic nickel, grade A, every increase of reduction by 10 per cent lowered the recovery temperature by 40 degrees Cent.

Before entering the temperature range of pronounced softening, a more or less pronounced increase of hardness induced by rolling was observed (1), (25). This phenomenon is the more pronounced the less pure the nickel, but it could not be proved beyond any doubt that this effect is completely absent in the highly pure carbonyl nickel preparations.

In view of the considerable shifting of the recovery points of carbonyl nickel with progressive cold working, and since it has been found that in melted material the recovery point is independent of the initial thickness before rolling, the experimental technique aiming at the scientific establishment of the true recovery temperatures of uncontaminated powder metals should be conducted in this manner:



Elimination of oxygen of the compressed powder in hydrogen at low temperatures, followed by sintering as close to the melting point as possible, repeated plastic deformations and recrystallizations at high temperatures until density determinations and preliminary recovery tests show no further changes. If this is the case, the "powder metal" may be considered to be structurally similar to metal which has passed through the liquid state but without having been contaminated by crucible walls or deoxidizers and without having undergone uncontrollable changes of the total balance of impurities. In this investigation, plastic deformations only by cold rolling have been applied. It is to be expected that hot working will considerably accelerate the conversion of powder metals into solid material.

### RESULTS

1. As a new approach to the study of the effect of contaminations upon the recovery of highly pure metals, metal powders were utilized.

2. The effect of small additions of silicon, tin and copper upon the recovery of carbonyl nickel was investigated.

3. Tin profoundly affects the recovery, 0.25 per cent tin shifting the recovery range upward by more than 200 degrees Cent. Additions of 0.25 and 0.75 per cent silicon cause a shift of 100 and 140 degrees Cent. respectively. The action of copper was mild, 0.75 per cent copper raises the softening temperature of severely worked carbonyl nickel by only 50 degrees Cent.

4. It has been confirmed that severely worked nickel (melted carbonyl nickel, electrolytic nickel) recovers from work hardening between 300 and 400 degrees Cent. and not between 400 and 500 degrees Cent. (Ransley and Smithells, Fetz). On prolonged heating, melted carbonyl nickel recovers from a 90 per cent reduction below 300 degrees Cent.

5. The recovery range may be lowered to 200-300 degrees Cent. (30-minute anneal) in severely worked, as-deposited electrolytic nickel and in carbonyl nickel powder which has not undergone a sufficient number of plastic deformations and annealing treatments. The latter material softens below 200 degrees Cent. on prolonged heating.

6. The recovery of the melted carbonyl nickel conforms with van Liempt's law concerning identical stages of recovery from cold work.

The sintered carbonyl nickel preparations follow a different time-temperature law of recovery.

7. A comparatively mild action of the speed of deformation (rolling) on the recovery was found. In melted nickel the initial thickness exerts little or no influence upon the recovery. An effect of the gas atmosphere (nitrogen, hydrogen, vacuum) during the annealing prior to plastic deformation could not be proved with electrolytic nickel.

8. The previously found law that the recovery points are directly proportional to the amount of cold work has been confirmed in all 12 cases investigated.

9. The extension of the recovery range of nickel cold-rolled between 10 and 90 per cent varies considerably. The nickel preparations made from powder showed the narrowest recovery range of 12.3 degrees Cent. per 10 per cent reduction and one of the two grades of electrolytic nickel employed exhibits the widest range of 40 degrees Cent. per 10 per cent reduction.

#### ACKNOWLEDGMENT

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## DISCUSSION

**Written Discussion:** By E. P. Polushkin, consulting metallurgical engineer, 500 W. 111th St., New York City.

In this second paper on the recovery of cold-worked nickel Mr. Erich Fetz has made another contribution of value. It has been my privilege to discuss his first paper, and now I wish to emphasize again the importance of one factor that has undoubtedly a marked influence on the recovery of cold-worked metals on annealing. It may explain, perhaps, the wide divergency of the results obtained by different investigators in this particular case. It is certain



that the effect of cold work on physical and chemical properties of metals depends not only on the amount of work applied to the metal, but also on the specific kind of the work. The degree of cold reduction is not a sole criterion of the inner changes produced in the metal by a deforming force. If we compare two identical pieces of metal with same reduction but reduced by two essentially different processes, one piece may be found on the verge of rupture while the other still capable of consuming a large volume of further deformation without failure. Such difference in the behavior of the same metal has been commonly found in industrial practice, in the processes employed for cold reduction of tubes and strips. Obviously, a piece of metal reduced by cold rolling is not the same as when reduced by cold drawing, and its response to annealing would be different. This may account, at least in part, for a difference of the result, obtained by Ransley and Smithells, on one hand, and the author in his latest work, on the other. The former investigators had been working with cold drawn nickel wires while the author with cold-rolled nickel strips.

One of the interesting features of the present contribution of the author is that the material used in his experiments was synthetically made, i.e., by compressing and sintering nickel powder of 325 mesh together with special additions. This was done in order to avoid contamination of the pure metal during melting. The author deserves a full credit for this new method of investigating the effect of impurities in metals. But if we wish to compare his results with the results of other investigators, the special way of manufacturing of his metal should be taken into consideration because it might have influenced the recovery of the metal.

**Written Discussion:** By Fred P. Peters, assistant editor, *Metals and Alloys*, New York.

In recent years we have been forced repeatedly to revise our accepted figures for the physical properties of many metals, as the latter have been prepared in ever increasing purity. With some, e.g., zinc and copper, the ultra pure products are commercially available and are in considerable demand because of the greater practical usefulness conferred on each by its unusually high purity. Again, with iron, the ultra pure product is as yet not commercially produced, but ingot iron of 99.9+ per cent purity is a standard product with enhanced corrosion resistance and other properties ascribable solely to its lower index of impurities. With still others, e.g., chromium, beryllium, etc., the laboratory preparation of ultra pure samples has shown that these metals, commonly believed to be "brittle" by nature, are in reality reasonably ductile and may be drawn into wire or rolled into sheet if small amounts of certain impurities are eliminated. The long strides that "impurity metallurgy" has taken is indicated by the recent appreciation of the powerful effect of traces of adsorbed or dissolved gases and gas-metal compounds on the properties of otherwise "pure" metals. C. J. Smithells, to whose work Mr. Fetz frequently refers, has identified himself most consistently with this subject and has just added to his earlier book "Impurities in Metals," another volume, "Gases and Metals."

Mr. Fetz is to be heartily congratulated for his extended study of the

recovery temperature of extremely pure nickel, first, because he has added nickel to the list of metals concerning which we now possess, through concerted improvement in purity, new knowledge or respect, and second, because his research has established the correct conditions and procedure for recovery studies in general. This particular contribution presents such a mass of data that discussion is not easy. Mr. Fetz's results have considerable scientific value in the changes in "the record" they involve: establishment of a new, much lower recovery temperature, and of the relative location of the recovery ranges of nickel of different origin. The results, however, have definite practical merit also, which it seems the author has not sufficiently emphasized. For example, confirmation of the previously derived law that recovery temperatures bear a linear relation to the amount of cold work is of great importance, for it permits practical determination in any case of the minimum softening temperature corresponding to a given amount of cold reduction. Also of considerable interest is the fact that electrolytic nickel could be successfully cold-rolled 90 per cent from the as-deposited state without any annealing treatment. What has happened to our legendary "hydrogen embrittlement" supposed to be characteristic of electro-deposited nickel? The application of metal powder technique to research of this nature is also worthy of note, and the significant differences in recovery behavior of the samples prepared from sintered powder and from melted powder are particularly important.

Inasmuch as Mr. Fetz's results depend largely on temperature measurements, a more detailed description of his temperature-measuring equipment might increase the value of his paper. Also, on pages 971 and 972 of the paper, the effect of atmosphere on the recovery of two grades ("A" and "B") of electrolytic nickel is discussed. The results on the two materials will be more significant if the author can give us some idea of the fundamental difference, either in analysis or structure, between grades "A" and "B."

In his first paper on this subject, presented last year, Mr. Fetz offered some data on grade "B" electrolytic nickel that are not in agreement with the results reported in this paper. Last year Mr. Fetz found that electrolytic nickel, grade "B," cold-rolled 80 per cent, recovered at 440 degrees Cent.; the same metal, annealed after deposition and cold-rolled 80 per cent, recovered at 454 degrees Cent., or at approximately the same temperature. This year, however, Mr. Fetz reports that electro-deposited nickel, Grade "A," cold-rolled 75 per cent from the "as-deposited" state without annealing, recovers at 255 degrees Cent. and cold-rolled 90 per cent recovers at 238 degrees Cent. On this basis, his 1937 material cold-rolled 80 per cent without previous annealing would recover at about 245 degrees Cent., which is approximately 200 degrees Cent. lower than last year's findings. This is a large discrepancy, although probably of minor importance, and any clarification Mr. Fetz can give will be greatly appreciated.

**Written Discussion:** By Don M. McCutcheon, metallurgist, Chemical and Metallurgical Laboratories, Ford Motor Company, Dearborn, Mich.

Mr. Fetz's paper on the recovery of cold-worked nickel is greatly appreciated. The results presented are of particular interest to anyone who has had the opportunity to observe the apparent anomalies in the recovery range of variously treated nickel samples.

The Ford Motor Company is interested in the production of a ductile and tightly adherent nickel electro-deposit as a base for chromium plated parts. A preliminary investigation has been made on the structural variations encountered in various nickel electro-deposits. The variations observed in the three test samples and their recovery curves are shown in Fig. 1. A commercial, annealed nickel sheet was used as control and shows a recovery temperature after cold-working, agreeing with the results of Wilson and Thomassen,\* in their Fig. 8, page 778. A ten minute vacuum anneal was used.

The curves in Fig. 1 show the effect of annealing temperature on the broadness of the X-ray diffraction line. A highly precise back-reflection technique

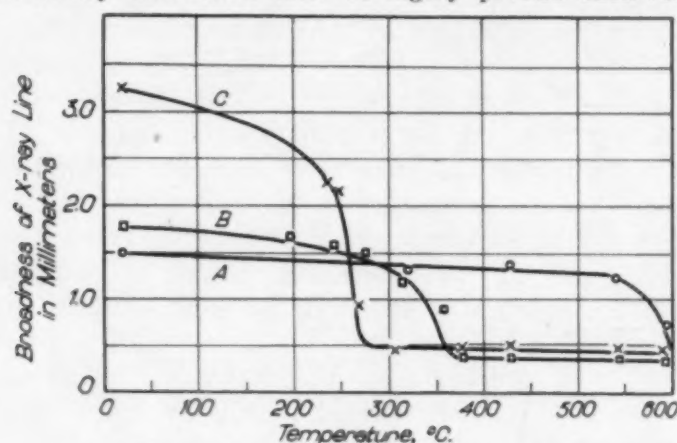


Fig. 1—Effect of Annealing Temperature on Broadness of X-ray Diffraction Line.

was used allowing a calculation of the grain size, approximation of the grain shape, and a rough calculation of the internal stresses in the plated metal. The grain size of the three samples as plated or cold-worked are as follows:

1. Curve-A, Commercial nickel, cold-worked...48 unit cells per grain
2. Curve-B, Normal nickel, ductile.....42 unit cells (plate)
3. Curve-C, Normal nickel, brittle.....20 unit cells (plate)

A calculation and comparison of the internal stresses in the nickel plate of ductile and brittle variety showed no difference. A value of approximately 20,000 pounds per square inch, was obtained and it was concluded that internal stresses played only a minor part if any, in the difference in hardness of the two samples. From an examination of the curves it may be observed that the smaller the grain size, the lower the recovery temperature for nickel plates produced in the same type of bath. The very slight difference in grain size between the normal ductile nickel plate and the cold-worked commercial nickel does not seem to account for the great change in recovery temperature from 1080 to 630 degrees Fahr. Various investigators have stated that hydrogen does not appreciably dissolve in nickel plate. Then what is the essential cause of the great decrease in recovery range as shown by curves in Fig. 1?

A detailed investigation of nickel electro-plate is in progress and it is

\*J. E. Wilson and L. Thomassen, "X-ray Line Broadness of Metals and Alloys and Its Relation to High Temperature Stability," *TRANSACTIONS, American Society for Metals*, Vol. 22, 1934, p. 778.



hoped that the structural differences causing the variations in hardness, grain size, orientation, and recovery range may be clarified. We are particularly interested in establishing a control of plating bath conditions and technique which will allow the production of the most favorable nickel plate for sublayers of chromium plated parts. The X-ray method, so far, seems to give us a method whereby the life of the plated parts in the salt bath may be predicted.

#### Oral Discussion

C. B. F. YOUNG:<sup>1</sup> While I was watching the data on the board, it occurred to me about this embrittlement, and any electro metallurgist will be interested in such a figure as was put there. The first discussor also mentioned that he wanted to know what became of it. After turning this over in my mind I happened to think that hydrogen released in aqueous solutions is in the atomic state, that is, H., while hydrogen used for annealing is H<sub>2</sub>, that is, molecular hydrogen, so undoubtedly that must be the answer.

Speaking of brittle nickel caused by hydrogen, any electroplater knows that nickel, deposited from a solution having a pH of 5.5 will not be brittle. He also knows if the pH is lowered to 4.0-4.5 a very brittle deposit is obtained which will curl of its own accord. However, I have just published some work in this field which appeared in the *Transactions* of the American Electrochemical Society, last week, in which it was found that if the pH is lowered to 3, brittle nickel is not obtained. This must be due to the fact that the hydrogen is not liberated in the nascent state or the nickel-hydrogen compound is not formed at this low pH. This might be of interest to some of you.

#### Author's Reply

I should like to express my appreciation of the valuable discussions presented by Messrs. Young, McCutcheon, Polushkin and Peters. Mr. Young's information on the occurrence and the effect of hydrogen in electrolytic nickel in contradistinction to ordinary nickel annealed in a hydrogen atmosphere is of interest.

Mr. McCutcheon's testing results do not refer to the recovery from cold working in general or to the recovery of work hardness in particular. Nevertheless his contribution will help us to understand recovery phenomena encountered in cold-rolled nickel in the electro-deposited state. Since the state of the metal prior to cold working appears to be of great importance, any light that is shed on changes of physical properties on annealing—I would not call it "recovery"—of nickel in the electro-deposited state is of interest, whether these changes are studied by hardness determinations, X-rays or the microscope. It is very interesting to learn that the X-ray lines may sharpen at temperatures as low as two hundred and sixty degrees centigrade, and that this sharpening takes place at a temperature which is the lower the finer the grain. It would be an interesting experiment if Mr. McCutcheon would apply some plastic deformations to his electro-deposited nickel and then determine where the hardness recovers. This "recovery" or loss of hardness would represent a change of two hardness effects, i.e., of the work hardness superimposed on the electro-deposition hardness. Attention may be called to the

<sup>1</sup>Columbia University, New York.

microscopic examinations of Jenicek showing that there is no correlation between the softening and microstructural changes in electrolytic nickel annealed as deposited. Grain size changes at temperatures which exceed the softening temperature range considerably.

Mr. Polushkin mainly raises two important questions: In the first place, is the difference in the method of cold-working, e.g., rolling versus drawing, partly responsible for the different behavior in plastic deformation and recovery therefrom; and secondly, are the results obtained on synthetic powder metals comparable with those obtained on metal that has passed through the liquid state?

Regarding the first question, Mr. Polushkin believes that a "piece of metal reduced by cold rolling is not the same as when reduced by cold drawing and its response to annealing would be different." I have found in the international literature no test referring to a comparison between the recovery of metallic material deformed by both methods. Lacking this fundamental experiment, an answer appears to be of a somewhat speculative nature, and I should like to maintain an open mind, although the various conceptions involved in this interesting problem may be touched upon. Many cases are known where greater reductions and plastic deformations in drawing can be applied than in cold rolling. However, whether and how the exhaustion of further plastic deformation is connected with recovery phenomena has to be experimentally proved. There may be an intimate relationship between the capacity for plastic deformation and the occurring strain hardening effects, particularly at the surface. However, we should be reminded that in different materials there is no simple relationship between work hardening effects and recovery therefrom. For instance, the greatest strain hardening effect took place in the carbonyl nickel containing 0.25 per cent tin. Yet this material recovers at a temperature almost 300 degrees Cent. higher than insufficiently wrought carbonyl nickel powder which exhibits considerably smaller strain hardening effects.

It is assumed that the total energy stored in the atoms of the powder samples is considerably larger than in melted material and that the hardness is not a quantitative indicator of the latent energy stored in the worked specimens. The exhaustion of plastic deformation seems to be a problem largely connected with the slip planes. If we take two identical pieces of metal, according to Mr. Polushkin's suggestion, and submit these to two different methods of cold working and if one test has to be discontinued at an early stage because of rupture (say in rolling) while the other sample can be still deformed (say by drawing) then both materials cannot be compared directly as they have undergone a different percentage of deformation. It is obvious that further reductions would be stopped on any work in the plant at the appearance of the first cracks. However, isolated edge cracks do not necessarily require discontinuation of recovery tests as long as a sufficiently great amount of sound material remains to determine its softening on annealing. If this procedure is not desired, extrapolation of the straight line plot of  $T_r$ /annealing temperature should answer the question whether samples of the same material, cold-rolled on one hand and drawn on the other to the same percentage, recover in similar fashion. Another way of establishing the entire softening temper-

ature range would be to employ smaller test pieces for the less suitable cold working method. It is generally known that samples of a smaller initial cross sectional area or of a smaller thickness lend themselves to greater reductions than larger samples. It was found in this investigation on samples of different initial thicknesses, that, in spite of the greater displacement from the original position or the greater flow in the heavier specimens, no difference in the recovery could be found with reference to identical percentages of reduction (rolling). Whether the higher recovery temperatures found by Ransley and Smithells are, therefore, due to their different method of cold working (that is, drawing instead of rolling) must yet be experimentally proved. One factor should not be overlooked, namely that their annealing times of five minutes are unusually short. Most investigators apply heat treatments of thirty minutes duration.

By connecting the recovery phenomenon with other physical properties which are indicative of the total energy stored in cold-worked metals, we may ultimately arrive at true or absolute recovery temperatures. The resistance to flow, or the limited ability to gliding along certain crystallographic planes in certain working methods may be comparatively loosely connected with the recovery phenomenon. The vibration amplitude of the atoms in cold-worked metals appears to be of greater significance. The vibration amplitude measured, for instance, by injections of radioactive matter, manifests itself in diffusion and recrystallization and as will be shown in a paper soon to be published, recrystallization of cold-worked nickel of different origin actually coincides with hardness recovery.

This leads to Mr. Polushkin's second question, regarding the effect of the "special way of manufacturing powder metals" on the recovery. Before embarking upon such a radical departure from the conventional method of recovery testing, naturally some preliminary experiments were carried out to study the feasibility of the new approach. As to details, I must refer you to the testing results which will appear soon in "*Revue de Métallurgie*." There a comparison is made between melted carbonyl nickel on one hand and compressed powder samples of pure carbonyl nickel submitted to different pretreatments on the other hand. And I can assure Mr. Polushkin that his apprehension that there "might be some differences" is quite justified. It may be said, in summation, that these differences are the greater the less the resemblance between the synthetic powder metals and the melted material. By applying a great number of plastic deformations and long-time intermediary annealings at high temperatures this conversion may be made complete. Density determinations are a valuable aid in checking the progress of the conversion and preliminary recovery tests would indicate whether the recovery range is shifted by further plastic deformations.

I want to thank Mr. Peters for his interesting bird's eye view comment on the complicated problem of recovery of pure metals in general and on the results of this investigation in particular. As to his three questions, I appreciate the importance of the temperature measurement. Every metallurgist who has bothered to determine the temperature gradient along the whole length of a muffle furnace and the possible temperature difference between a thermocouple suspended in the furnace atmosphere and samples in contact with the



furnace walls, is fully aware of this possible source of error. In this investigation, a copper sleeve of 5-inch length,  $2\frac{1}{4}$ -inch outside diameter and 1-inch bore was employed. Its temperature was simultaneously measured three times, i.e., by a calibrated, shielded Pt-Pt/Rh thermocouple, an unprotected chromel-alumel couple and as far as possible, by a high temperature Hg-thermometer, all resting on the copper sleeve. The readings of the calibrated Pt-Pt/Rh couple were used and they checked the other readings very closely after sufficiently long stabilization times. The electric furnace was controlled by an unprotected thermocouple made of thin chromel-alumel wires placed close to the resistance winding at the outside of the muffle. By this arrangement the temperature variations inside the muffle and particularly in the copper block, which weighed over 5 pounds, became negligible. A very small amount of metal is necessary to determine the hardness after the 30 minute annealing treatment. All samples to be tested at the same temperature were placed in the furnace at the same time. They rested in a small trough made of nickel foil of 0.004-inch thickness. The bottom of the trough fitted the copper sleeve. Due to (1) this intimate contact, (2) the relatively small amount of metal to be brought up to temperature and (3) the great heat capacity of the copper block, a rapid heating to the testing temperature was secured.

Regarding the two grades of electrolytic nickel, called "A" and "B," I am unable to state the correct analysis of the commercial "A" grade. The most recent typical analysis of the same supplier is given by E. M. Wise (*Transactions*, Institute of Radio Engineers, Vol. 25, June 1937, p. 714) as follows:

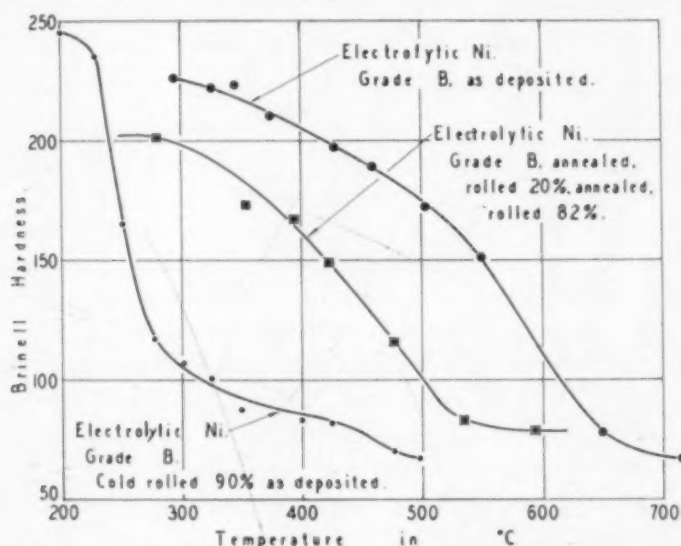
99.95% Ni + Co  
0.01% Cu  
0.01% Fe  
0.001% S

Regarding the refined electrolytic nickel, designated as "B," this is the highly pure nickel, which Professor Jette and his collaborators are using in the high precision X-ray work at Columbia University. The analysis given in all publications of the X-ray Department shows 0.11 per cent cobalt, 0.013 per cent iron and traces of silicon and sulphur. Spectroscopic analysis failed to disclose the presence of appreciable amounts of other metals. Both grades are apparently of a rather high purity, which is corroborated by their low recovery temperatures. The slight differences in the recovery of annealed samples are, as I believe, not due to the slight differences in the chemical analysis, but are due to differences in structure. The significance of the structure in recovery tests has not been fully recognized. However, a discussion of this important factor would transcend the scope of this oral reply. The difference in the recovery of the electrolytic nickel "B" in the vacuum-annealed state and annealed in nitrogen, is that the former recovers from a 10-90 per cent reduction in the range of 446-632 degrees Cent. while the latter recovers between 442 and 559 degrees Cent. In other words, the severely worked samples recover in the same manner while there is a discrepancy at smaller plastic deformations. However, this difference, supposedly brought about by structural effects which may be due to the furnace atmosphere, is comparatively unimportant considering the low recovery temperature of 238

and 225 degrees Cent. of electrolytic nickel, grade "A" rolled 75 and 90 per cent respectively in the as-deposited state.

I am indebted to Mr. Peters' written discussion calling attention to the fact that the "B" grade was reported last year to show no great difference in recovery, whether it was cold-worked in the "as-deposited" state or in the cold-worked and recrystallized state. A retest has been made by rolling a sample of "B" grade 90 per cent as electro-deposited. It was found to lose 50 per cent of its hardness at 250 degrees Cent. thus supporting the hypothesis that the fine grain is responsible for the low recovery point. The new softening curve of electrolytic nickel, "B" grade, in the electrodeposited state and cold-rolled as deposited is graphically presented below.

Referring again to Mr. Peters' third question, in analogy to the softening tests on electrolytic nickel, "A" grade, presented in Fig. 20 of the paper, the experiments were repeated using the "B" grade. The results are presented in the curves below. The losses of 50 per cent of initial hardness—be it due to



electrodeposition, cold-working or electrodeposition plus cold-working—in relation to pretreatment of both grades of electrolytic nickel are compared:

	"A" Grade	"B" Grade
Pretreatment .....	T <sub>r</sub> in °C. (Loss of 50% of Initial Hardness)	
As deposited .....	508	550
90 per cent rolled as deposited .....	238	250

Both materials apparently behave qualitatively in the same manner, i.e., recovery takes place at much lower temperatures than observed in severely cold-worked, melted metal of highest purity. The electrodeposited nickel, "B" grade, softens at somewhat higher temperatures but the recovery from cold work is shifted to higher temperatures in comparison with the "A" grade.

The formerly presented recovery curve S, Fig. 1<sup>1</sup> of supposedly electrodeposited nickel, "B" grade, must be withdrawn for although the high initial hardness of 250 Brinell of the 82% cold-rolled sample would indicate that the

<sup>1</sup>E. Fetz, "Recovery of Cold-Worked Nickel on Annealing," TRANSACTIONS, American Society for Metals, Vol. 25, 1937, p. 1036.

as-delivered material was in the electrodeposited state, it seems impossible that this was so. Highly pure nickel does not attain such cold work hardness even on severe plastic deformations. The earlier tests were carried out in 1935 and unfortunately no structural investigation of both samples of "B" grade in the electrodeposited state can be performed at this time. In the light of unpublished experiments, the effect of structural differences on the location of the temperature range of hardness recovery is unexpectedly great.

Attention may also be called to recovery tests by Wazau<sup>2</sup> on electrolytic nickel in the electrodeposited state. Although Wazau employs the drastic method of cold-working by upsetting and in spite of the fact that unusually long annealing times of 5 hours were employed, the temperatures of recovery of hardness (due to electrodeposition plus cold-working) are rather high and not very much different from ordinary, recrystallized nickel:

Deformation (by upsetting)	Recovery temp. T.
10%	615 degrees Cent.
14%	575 degrees Cent.
20%	570 degrees Cent.

The objection may be raised that the maximum amount of cold work applied by Wazau was only 20 per cent and attention may be called to those cases where the recovery temperature of nickel was lowered 40 degrees Cent. by every 10 per cent increase in deformation by rolling. (See Figs. 7, 8 and 9). This would open the possibility that if Wazau had cold-worked his electrodeposited sample 90 per cent, it might have recovered at some 310 degrees Cent. However, unpublished experiments of the present author yielded an unusually narrow recovery range of electrodeposited nickel cold-rolled 30-90 per cent. As a matter of fact, a sample cold-rolled 30 per cent in the electrodeposited state lost half of its maximum hardness at slightly over 300 degrees Cent. In view of this contradictory evidence, more research on the nature and behavior in recovery tests of electrolytically deposited metals is desirable.

It appears that light should be shed on the state of the metal prior to cold working and in this regard experimental data such as presented by Mr. McCutcheon are very valuable. Minute details on the experimental technique should be presented. For instance information on Mr. McCutcheon's method of "rough calculation of the internal stresses in the plated material" would be of interest. At present the interpretation of the X-ray picture of cold-worked metals is under careful scrutiny.<sup>3,4</sup> As to the X-ray picture of electrodeposited nickel and the changes on annealing, Mr. McCutcheon's interpretation of the underlying cause would be of interest. Supplementary tests on the changes of other physical properties (e.g. density, hardness, grain size, hydrogen content, etc.) during annealing appear to be very desirable. In the light of previous experimental evidence, recrystallization, softening and volume changes of electrodeposited nickel take place at much higher annealing temperatures than were found by Mr. McCutcheon for the sharpening of X-ray diffraction lines.

<sup>2</sup>G. Wazau, "Das Gefüge des Elektrolytnickels," *Zeitschrift für Metallkunde*, Vol. 16, 1924, p. 412.

<sup>3</sup>W. Boas, "Röntgenographische Untersuchungen der Gitterstörungen in deformiertem Gold," *Zeitschrift für Kristallographie*, Vol. 96, 1937, p. 214.

<sup>4</sup>W. Boas, "Ueber Röntgeninterferenzen an Gestörten Gittern," *Zeitschrift für Kristallographie*, Vol. 97, 1937, p. 354.



## THE EFFECTS OF LONGITUDINAL SCRATCHES ON VALVE SPRING WIRE

BY F. P. ZIMMERLI, W. P. WOOD AND G. D. WILSON

### *Abstract*

*The object of this study was twofold: the development of a method for the measurement of depths and contours of surface scratches upon spring wire, and secondly, to determine the effects of scratches with varying depths and contours upon the endurance limit in torsion of a typical steel spring wire. A special type of machine for carrying out torsional fatigue tests upon small sections of wire was designed and built. Scratches of controlled depth and contour were produced upon straight wire sections and their endurance limit in torsion compared with that of the same wire in the unscratched condition.*

*As would be expected, the presence of longitudinal scratches lowers the endurance limit of the wire to an appreciable extent. The decrease in endurance limit produced by the scratches was not, however, as great as the differences observed between polished and commercial wire when in the form of helical springs. The contours of the scratches were much more important than the depths of the scratches.*

*Mechanical defects such as scratches produced during drawing and coiling operations can never be as serious in causing spring failures as seams produced in the wire during the manufacture of the wire.*

THE presence of longitudinal scratches upon the surface of spring wire has always been troublesome to the manufacturers of helical springs. The cause, first considered, of all spring failures is a seam or scratch, yet all who have examined the metal adjacent to such failures are aware of how seldom the scratch or seam is present and how other reasons for the failure must be sought. The question is always in mind regarding the effects of depth and contour of surface scratches. Is there any limiting size or shape of scratch which may be considered relatively harmless as far as the

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. Of the authors, F. P. Zimmerli is Chief Engineer, Barnes-Gibson-Raymond Division of Associated Spring Corporation; William P. Wood is Professor of Metallurgical Engineering, University of Michigan; and G. D. Wilson is Metallurgist, Barnes-Gibson-Raymond Division of Associated Spring Corporation. Manuscript received April 14, 1937.

performance of the spring is concerned since it is well known that springs do perform in a satisfactory manner with a certain amount of surface discontinuity? No inspection methods have seemed to furnish a satisfactory answer to this question.

There are several causes to account for the presence of these scratches. They may appear during the processing at the wire mill, during the coiling operation, or may be the result of careless handling of the finished springs. The important thing, however, is not the original cause of the scratches, but what their potentialities are as a future source of trouble during the life of a spring.

The investigation reported in this paper was carried out with two aims in mind: First, the development of a method for accurately measuring the depths and contours of scratches, and secondly, to determine the effects of scratches with varying depths and contours upon the endurance limit in torsion of a typical steel spring wire. The investigation is in no wise considered as being complete, but is presented in the nature of a progress report with the thought that it may contain matters of interest and may stimulate helpful comment, since it is hoped that the study may be continued.

#### PREVIOUS INVESTIGATIONS

A considerable amount of work has been done on the general subject of fatigue. The greater amount of attention has been given to bending—not so much to torsion. A study of the literature shows that little attention has been given to the effects of surface discontinuities upon the endurance limit in torsion. Many papers, of course, have been written which have a bearing directly or indirectly upon the subject. Space does not permit of a complete review of the literature. A selected bibliography is appended to this paper and, of course, some references from this list will be used during the discussion. In this bibliography, the first eight references are concerned with the development of the theory underlying the method used for calculating the stress at the bottom of a scratch on a bar which is subjected to torsional stress; the references from 9 to 13, inclusive, cover methods of measuring the depth and shape of scratches; and references 14 to 37, inclusive, cover the published work on the effect of surface discontinuities upon fatigue test results.

#### TEST PROCEDURE

*Torsion Fatigue Machine.* A survey of the torsional fatigue

machines on the market at the time of this investigation failed to locate one suitable for testing small wire specimens. Three torsional fatigue machines were therefore built after the design shown in Fig. 5. These machines are of the constant strain type. A movable jaw (A) oscillates through a rather small angle while the second jaw (B) is fixed. The test specimen is gripped securely by these two jaws. A connecting rod (D) is attached to a slotted rod (C) which is an integral part of the movable jaw (A). The other end of the

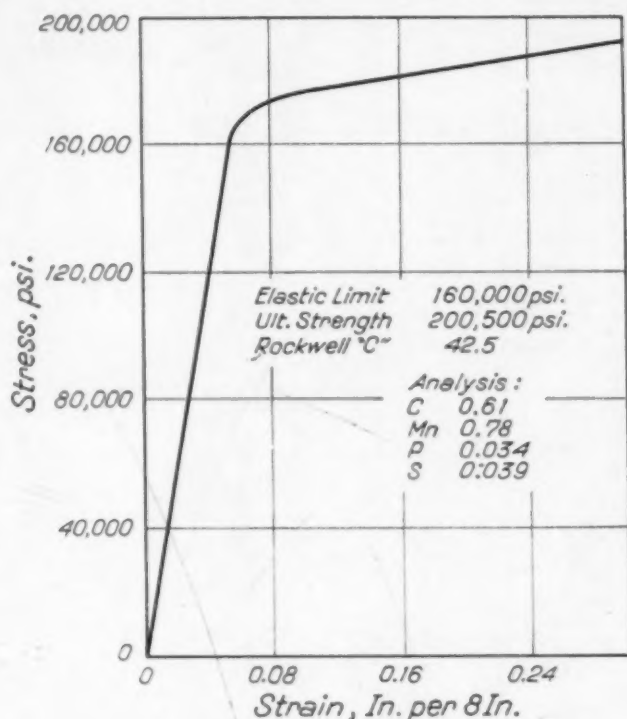


Fig. 1—Tensile Stress-Strain Curve. For 0.169 Inch Steel Wire Used in Fatigue Tests.

connecting rod fits over a variable crank mechanism which is actuated by electric motor (X). The angle of twist is regulated by the double eccentric (U and V) so that stress values may be varied from 0 to a maximum or from a minus maximum to a plus maximum. All tests in this study were run from 0 to a maximum, since that is the manner of stress variation in helical springs. 1725 stress cycles per minute were applied, the total number of cycles being indicated by a counter (Y).

The machine is stopped automatically when the test piece fails. A small current from a 6-volt battery passes through the test specimen as long as the specimen is unbroken. When the specimen fails, this current is interrupted and a relay is opened. This actuates an



alternating current relay which cuts off the motor. The current through the test piece is about 0.15 ampere.

### STEEL USED AND PREPARATION OF SPECIMENS

The steel chosen for this investigation was a high grade acid open-hearth valve spring wire. It was felt that such a material would be as nearly typical as anything. The chemical and physical properties of this wire are given in Figs. 1, 2, and 3. It is a tempered carbon

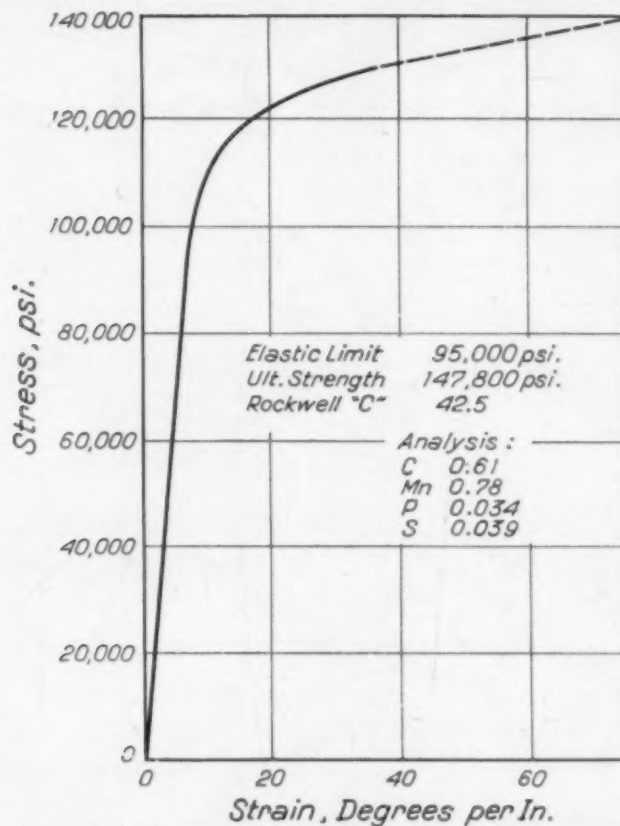


Fig. 2—Torsion Stress-Strain Curve for 0.169 Inch Steel Wire Used in Fatigue Tests.

steel wire produced in the United States and was taken from regular stock. Microscopical examination showed the wire to be free from seams and nonmetallic inclusions. The diameter was approximately 0.169 inch. Torsion tests were made on this material turned down to 0.110 inch, which was the diameter of the reduced part of the torsional fatigue test specimens. It will be noted from a comparison of Fig. 3 with Fig. 2 that there is no appreciable difference in the static torsional properties at this diameter and at the full diameter of the wire.

Fig. 4 is an isometric drawing of the device used for making scratches on test specimens. It consists of a holder for the tool and a device which holds the test specimen and moves it back and forth under the tool.

The tool holder (A) is hinged on a pin (B) and may be tilted back so that the tool can be easily removed for honing. The tool

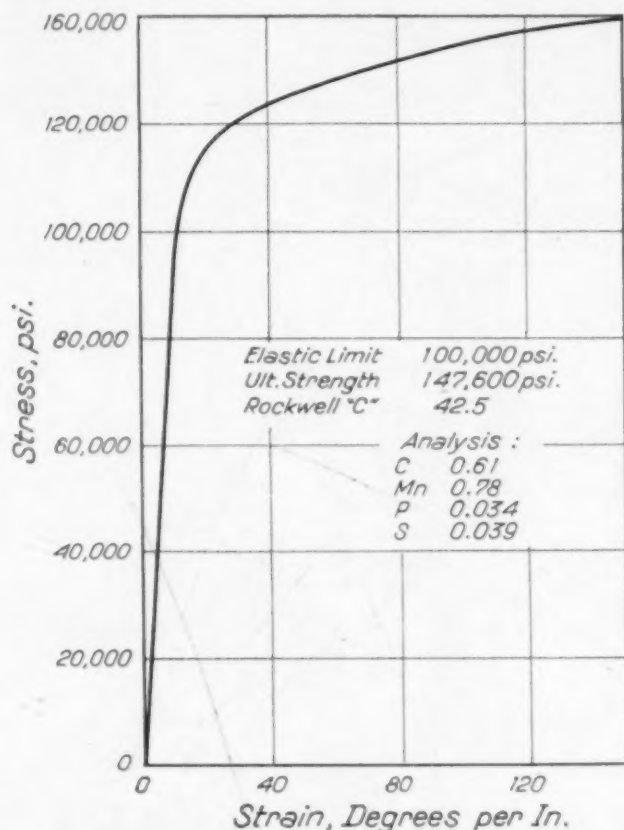


Fig. 3—Torsion Stress-Strain Curve for 2 Inch Section of 0.169 Inch Steel Wire Turned to 0.110 Inch Used in Fatigue Tests.

feed screw (D) is keyed to the tool by means of a key (E) and a slot (F) in the top of the tool. This enables the tool to be moved up and down with a positive action. The feed screw is graduated with 0.0005-inch divisions. These divisions are quite large so it is actually possible to move the tool up or down a distance of 0.00025 inch.

The device for holding the test specimen and moving it back and forth under the tool is essentially a moving stage (G) actuated by a screw and crank (H). The test specimen is held firmly on the stage in a direction parallel to the motion of the stage by two clamps

(J). The clamps are so designed that the test specimen (K) is always centered under the point of the tool.

Fig. 4 also includes a drawing of the test specimen as used in these tests. The reduced section is 0.110-inch diameter for a linear

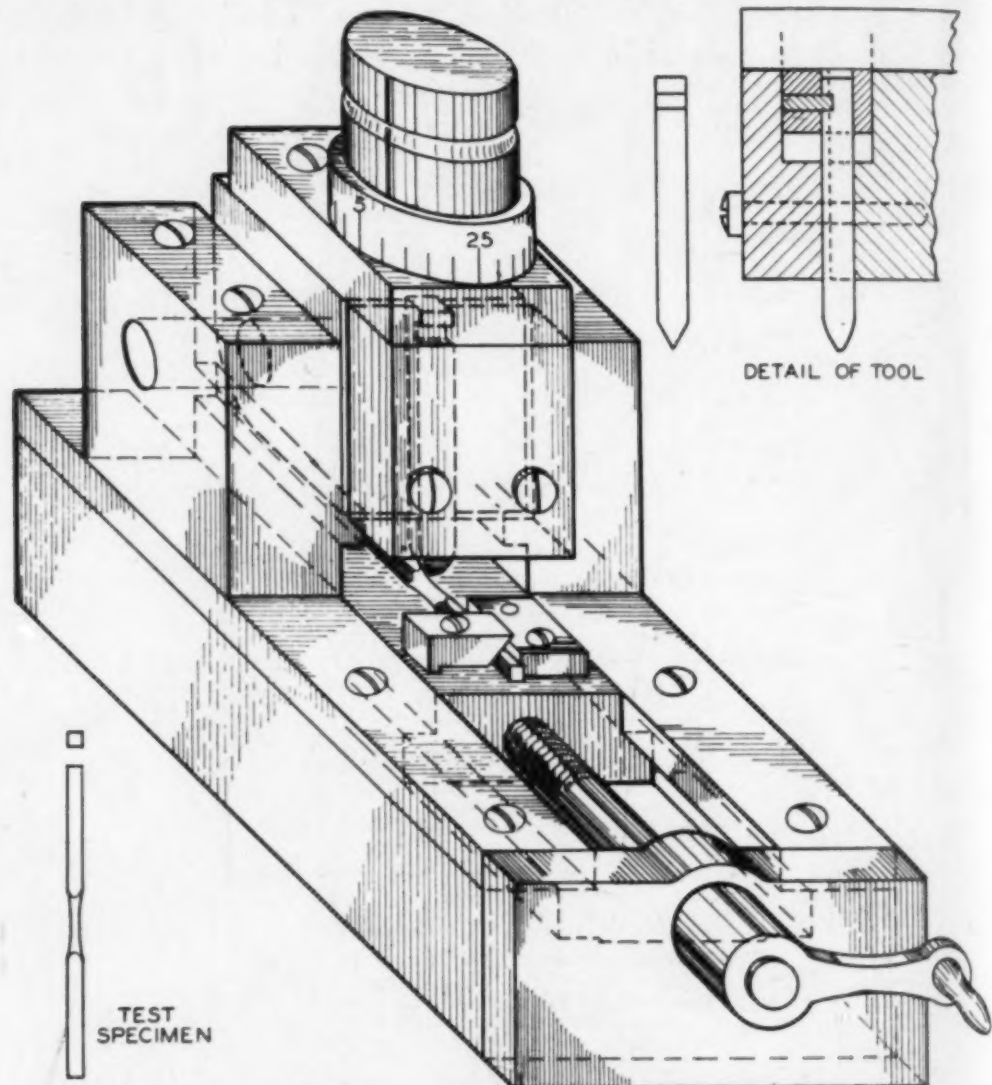


Fig. 4—Device for Making Controlled Scratches.

distance of 0.125 inch. The grip ends were ground to a square cross section about 0.134 inch on a side. All test specimens were ground in a lathe with 0, 00, and 000 emery paper. Finer paper was not used since H. F. Moore (21) shows no appreciable gain in endurance limit with the more highly polished test specimens. The diameters of the test specimens were carefully measured with pointed micrometers to within 0.0005 inch. The diameter used for calculat-

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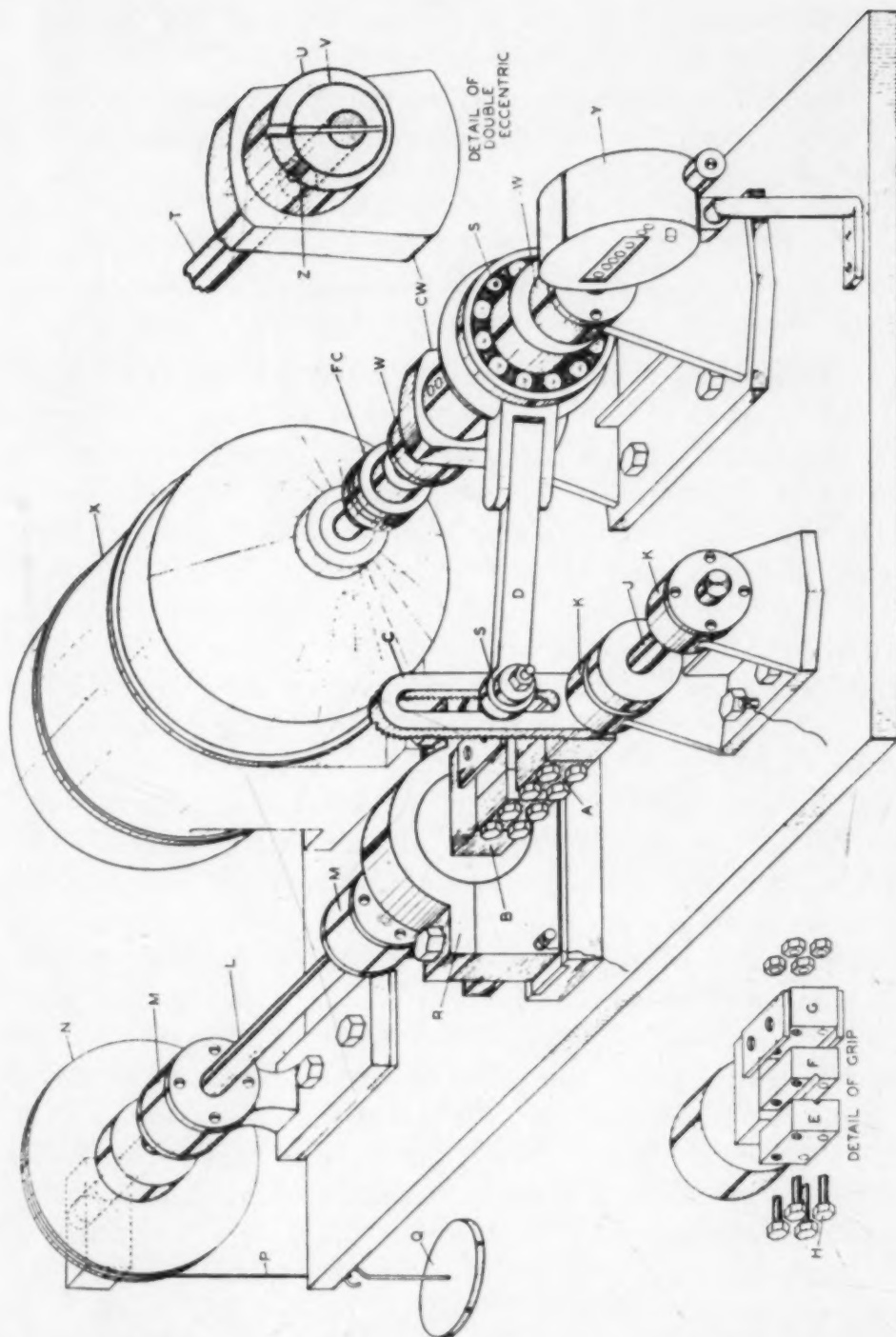


Fig. 5—Diagrammatic Sketch of Torsional Fatigue Machine.

ing stresses was the diameter of the smallest part of the reduced section. If the specimen was slightly out of round, the smallest diameter was used.

Before starting a test run, it was necessary to determine the angle of twist required for the desired maximum stress. The specimen was inserted in the fatigue machine and clamped in the grips. The stress present in a cylindrical rod under a twisting moment is given by the expression

$$C = 16 PR/d^3$$

where  $S$  = stress in outer fibres in pounds per square inch  
 $P$  = load in pounds  
 $R$  = length of lever arm in inches  
 $d$  = diameter of the rod in inches.

The twisting moment required to produce any given stress in the test specimen could be calculated since the diameter of the specimen was known. When the angle of twist corresponding to a given stress was determined in the fatigue machine, the twisting moment was produced by weights acting on the cable and pulley attached to the shaft of the fixed jaw. Knowing the radius of the pulley and the thickness of the cable, it is possible to calculate the lever arm through which the weights act. It is then possible to determine the weight necessary to produce the required stress in the specimen.

The angle of twist is measured by a system consisting of a mirror mounted on the fixed jaw shaft of the machine and a telescope and scale located a few feet from the mirror. The mirror was mounted on the shaft in such a manner that it rotated with the shaft about its axis. The rotation of the fixed jaw and shaft caused by a dead load on the cable produced a corresponding and equal rotation of the mirror. The load calculated for the given stress was suspended by the cable and a reading taken on the scale by means of the telescope which was provided with cross hairs. The load was then removed and another reading taken. The difference between these two readings gave a value of the angle of twist in centimeters on the scale. It was not essential that the angle be expressed in degrees or radians as it could be more conveniently expressed in centimeters on the circular scale. It was calculated that this method of measuring the angle of twist was accurate to about  $\pm 0.3$  per cent. This corresponds to about 0.016 degree or 0.96 minute.

Lead plates were used for the loads to determine the angle of twist. These loads were measured on a Chatillon set of scales which were accurate to about  $\pm 0.15$  ounce or  $\pm 0.1$  per cent. The two dead center positions of the crank of the fatigue machines were found by using an indicator on the movable jaw. The angle through

which this jaw turns while the crank moves from one dead center position to the other must equal the angle of twist as previously determined to give the required stress. The angle of twist of the movable jaw was varied by changing the position of the small end of the connecting rod on the slotted rod (C) in Fig. 4. When this procedure did not give a sufficient range of adjustment, the relative positions of the two eccentrics (U) and (V) were changed.

Preliminary tests showed that the fatigue test specimens invariably suffered a permanent angular deformation or set shortly after the fatigue tests were started. This effect was also noted by Swan, Sutton and Douglas (34). The amount of the set was determined as follows: The crank of the machine was set at the position where no stress should be present in the test piece. The position of the fixed jaw was then read by means of the mirror, telescope and scale. The jaw was then unclamped, which allowed it to come to a position where no stress was present in the test piece, and a second reading taken. The difference between these two readings represented the amount of permanent angular deformation of the test piece. This behavior of the test piece, of course, changed the stress cycle to which the specimen was supposed to be subjected. For example, if the original stress range was 0 to 100,000 pounds per square inch, after this permanent set had occurred the stress range might be found to be -15,000 to +85,000 pounds per square inch.

After some consideration, it was decided to compensate the effect of this permanent set by subjecting the sample to an initial twist (which produced an initial positive stress) before the fatigue test was started. This twist was found by trial to amount to from 5 to 25 per cent of the maximum twist used, varying directly with the size of the stress range. This initial twist was applied by dead loads and was measured in the same manner as the angle of twist corresponding to the stress range. Preliminary tests also showed that this permanent set took place during the first few hours of the test, the stress cycle remaining constant thereafter.

The machines were started and allowed to run for several hours after the initial twist had been applied to the test specimen. The machines were then stopped and the cranks set at the zero stress positions by means of the indicator previously described. Readings of the position of the fixed jaw before and after the clamp was loosened were obtained with the mirror and telescope. If the dif-



ference between the first and second readings was negative the test specimen had deformed more than was anticipated. In this case, an additional initial twist was applied to the test piece. If the difference between the two readings was positive, the deformation of the test piece was not as great as had been expected. If the two readings were identical, the deformation had been estimated correctly. In the last two cases, there was no need to apply additional twist to the test specimen, and the fixed jaw was therefore clamped at the zero stress position. This procedure was repeated until no further deformation in the test specimens was noted. In no case did it have to be repeated more than twice, unless as a precaution to make sure that the stress cycle had not changed. As mentioned previously, after the first few hours of the test, the stress cycle did not change appreciably. This was found to be true by repeating the above procedure at the completion of the test in cases where the test specimen did not fail.

Three specimens were tested at each stress for every type of scratch. The failure of two out of three specimens determined that the stress was above the endurance limit. If two of the three specimens did not fail, it was concluded that the maximum stress was below the endurance limit.

#### MEASUREMENT OF SCRATCHES

The tools used for making scratches were made of high speed steel, Stellite or diamond. The angles of the high speed steel tools were 15, 30, and 45 degrees. The Stellite tool and the diamond tool each had 30-degree angles. Although these tools had angles as stated, this gave no indication as to the contours of scratches made by the tools, for every time a scratch was made, a certain amount of the tool point would be worn away and the resulting scratch might have an entirely different contour than the original one of the tool. For the same reason, no accurate idea could be formed of the depth of the scratch, even though the tool was fed a definite distance into the test specimen. It was therefore necessary to devise a method to determine accurately the shape and depth of the scratches after they were made.

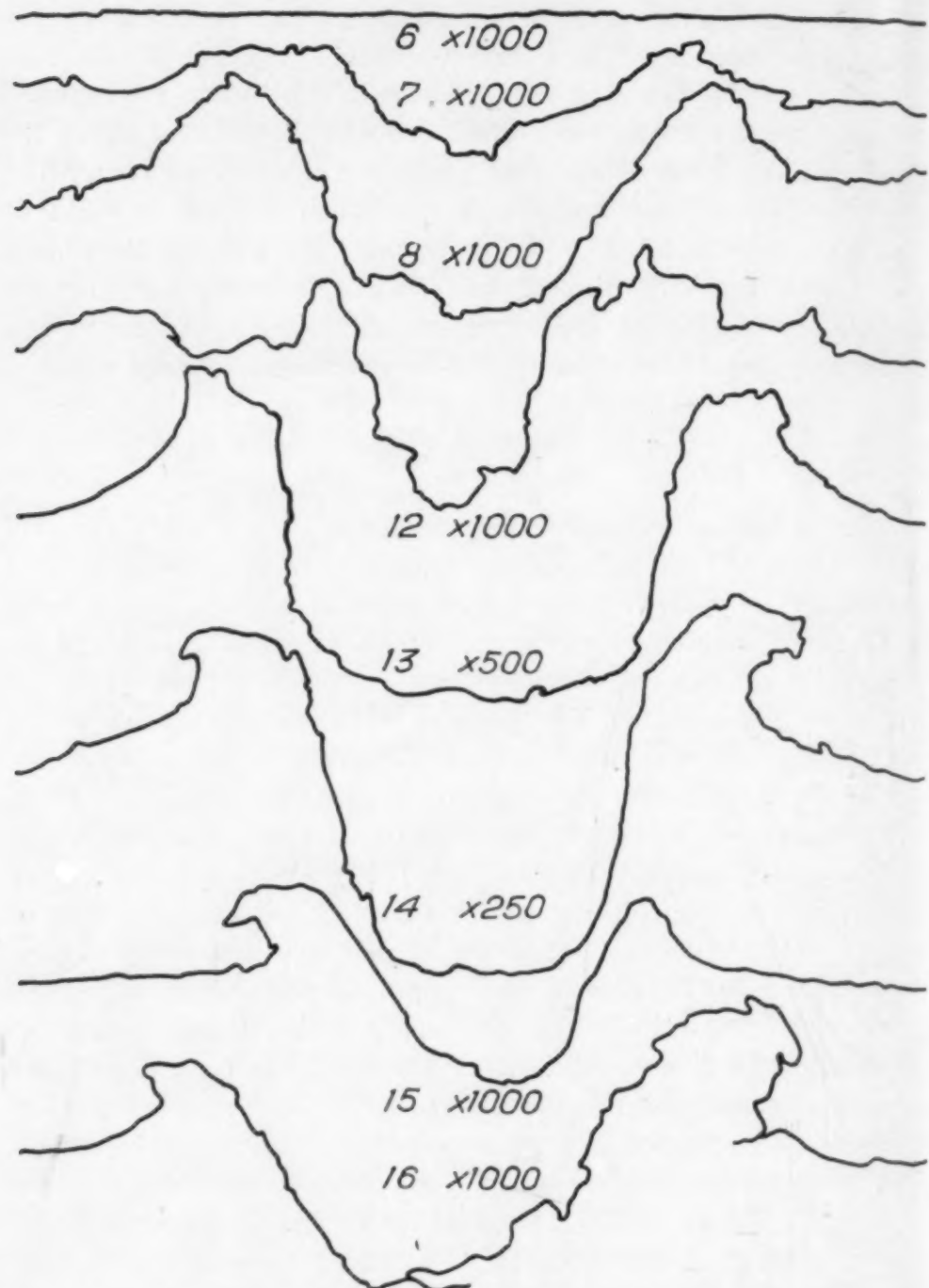
After trying several methods, the following one was adopted: The test specimen was cut in two about 0.06 inch from the point at which it was desired to examine the scratch. The piece of the

specimen was heavily plated with copper. The thickness of the copper plate was not less than 0.02 inch. The piece was then mounted vertically in a steel ring. About 0.06 inch was then ground off the ring and the specimen. This brought the point at which the scratch was to be examined very near the ground surface. The piece was then given a metallographic polish so that it could be examined under the microscope. Photomicrographs were then made of the scratch and from these it was possible to accurately study the contours and depths of the scratches. This same procedure was used to examine the scratches found on commercial spring wire.

### DISCUSSION OF RESULTS

The values for endurance limit obtained in this study are presented in conjunction with photomicrographs of transverse sections of scratches or contour lines traced from such photomicrographs. The intended depth and the actual depth of each scratch are indicated; also the per cent decrease in endurance limit caused by the presence of the scratch. The purpose in presenting results in this form is to make it possible to readily compare the contour and depth of the scratch with the actual endurance limit obtained. From the standpoint of this work, the contour of the surface shown in Fig. 6 may be considered to be ideal. The endurance limit of a test specimen having such a surface may be used as a standard of comparison for the endurance limits of test specimens having scratched surfaces. The endurance limit of test specimens with a surface contour like the one shown in Fig. 6 is 110,000 pounds per square inch. In all cases the endurance limit has been determined to the nearest 5000 pounds per square inch. In this work the endurance limit is defined as that stress which will allow the sample to just withstand 10,000,000 oscillations. Any stress above this will cause failure before 10,000,000 stress cycles have been carried out.

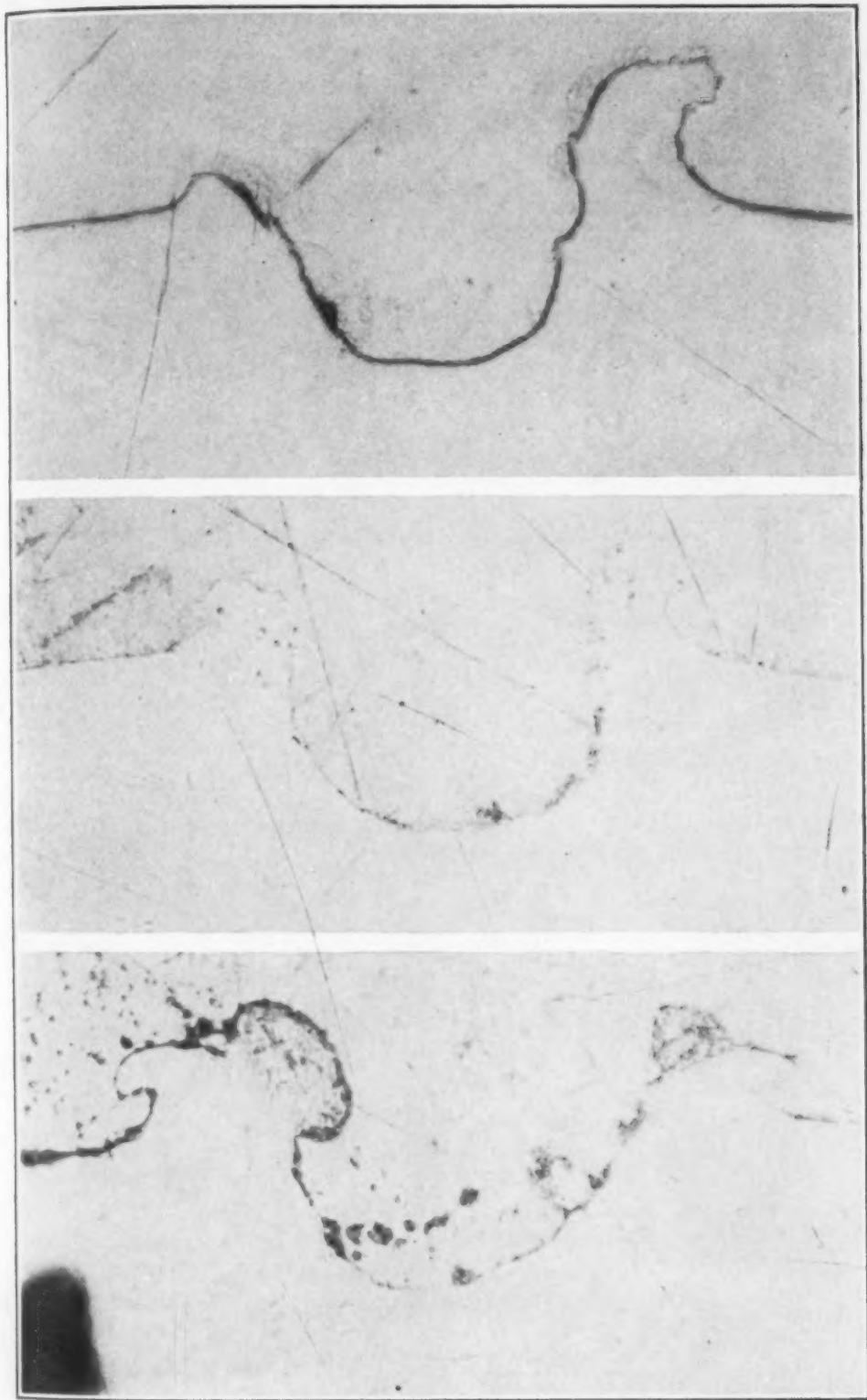
The endurance limits of test specimens scratched with high speed steel tools are given in Figs. 7 to 19, inclusive. It will be noted that most of these scratches have a rather large radius of curvature at the bottom of the scratch. The shape of the scratch does not vary greatly with the angle of the tool. The point of the 15-degree tool wore down faster than the others, and hence, the scratches produced by this tool did not lower the endurance limit greatly. The 30-degree tool produced the sharpest scratches. For



Lines Traced from Photomicrographs of Specimens Scratched in Varying Ways.  
Magnification Indicated on Each Curve.

Fig. No.	How Scratched	Intended Depth Inch	Actual Depth Inch	Endurance Limit Pounds Per Square Inch	Per Cent Decrease in Endurance Limit
6	Polished	.....	.....	110,000	...
7	15° H.S. Tool	0.0005	0.00034	110,000	0.0
8	15° H.S. Tool	0.001	0.00056	105,000	4.5
12	30° H.S. Tool	0.001	0.00071	100,000	9.0
13	30° H.S. Tool	0.002	0.0015	90,000	18.1
14	30° H.S. Tool	0.005	0.0042	90,000	18.1
15	45° H.S. Tool	0.0005	0.0005	110,000	0.0
16	45° H.S. Tool	0.001	0.00056	100,000	9.0





Photomicrographs of Specimens Scratched in Varying Ways.

Fig. No.	How Scratched	Intended Depth Inch	Actual Depth Inch	Endurance Limit Pounds Per Square Inch	Per Cent Decrease in Endurance Limit
9 (upper)	15° H.S. Tool	0.002	0.0013	105,000	4.5
10 (center)	15° H.S. Tool	0.005	0.0033	100,000	9.0
11 (lower)	30° H.S. Tool	0.0005	0.00063	110,000	0.0

Ways.  
Per Cent  
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0.0  
4.5  
9.0  
18.1  
18.1  
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scratches over 0.002-inch deep the 45-degree tool did not make as sharp scratches as the 30-degree tool.

In all cases it will be noted that the endurance limit does not decrease in a constant manner as the depth of the scratch increases. This is due to the fact that as the depth of the scratch is increased,

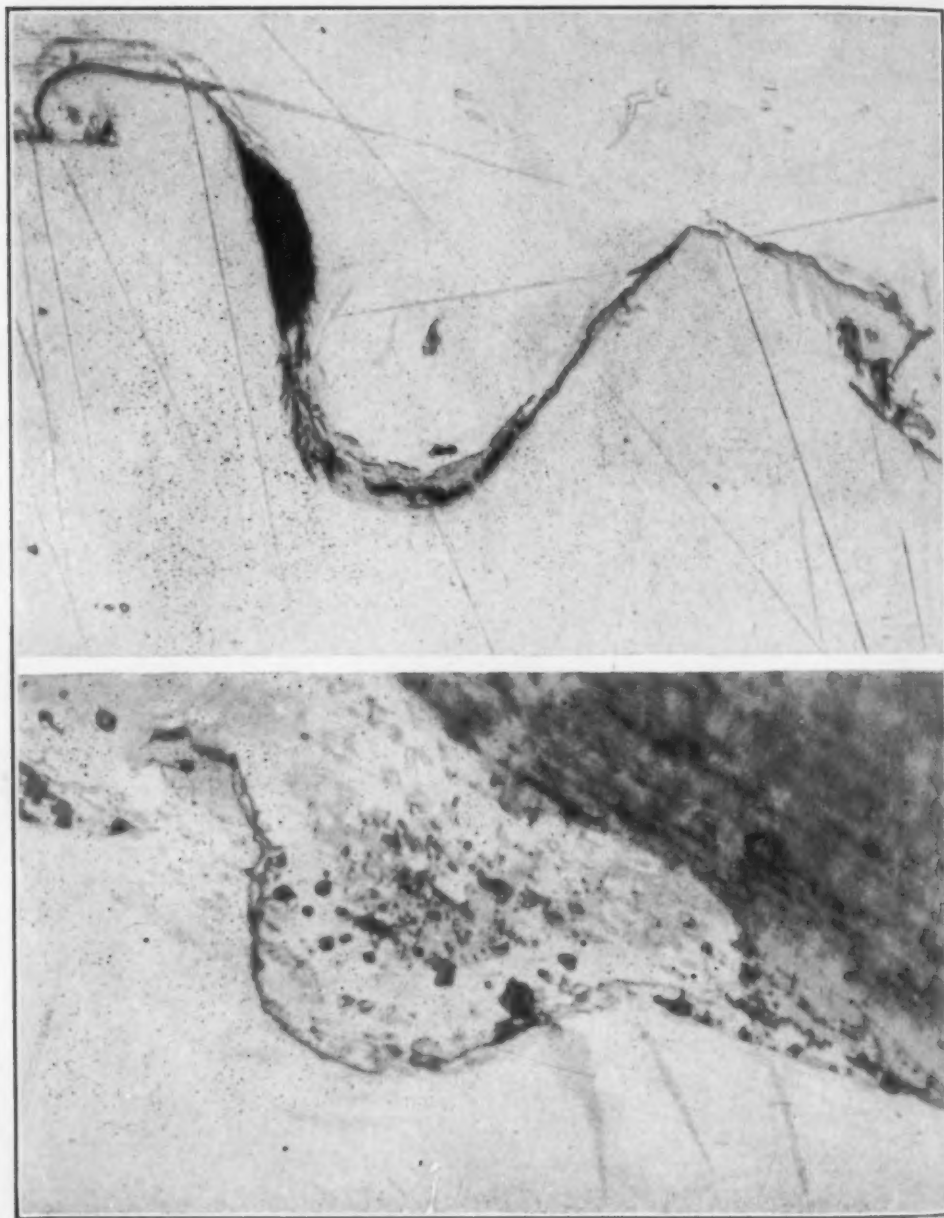
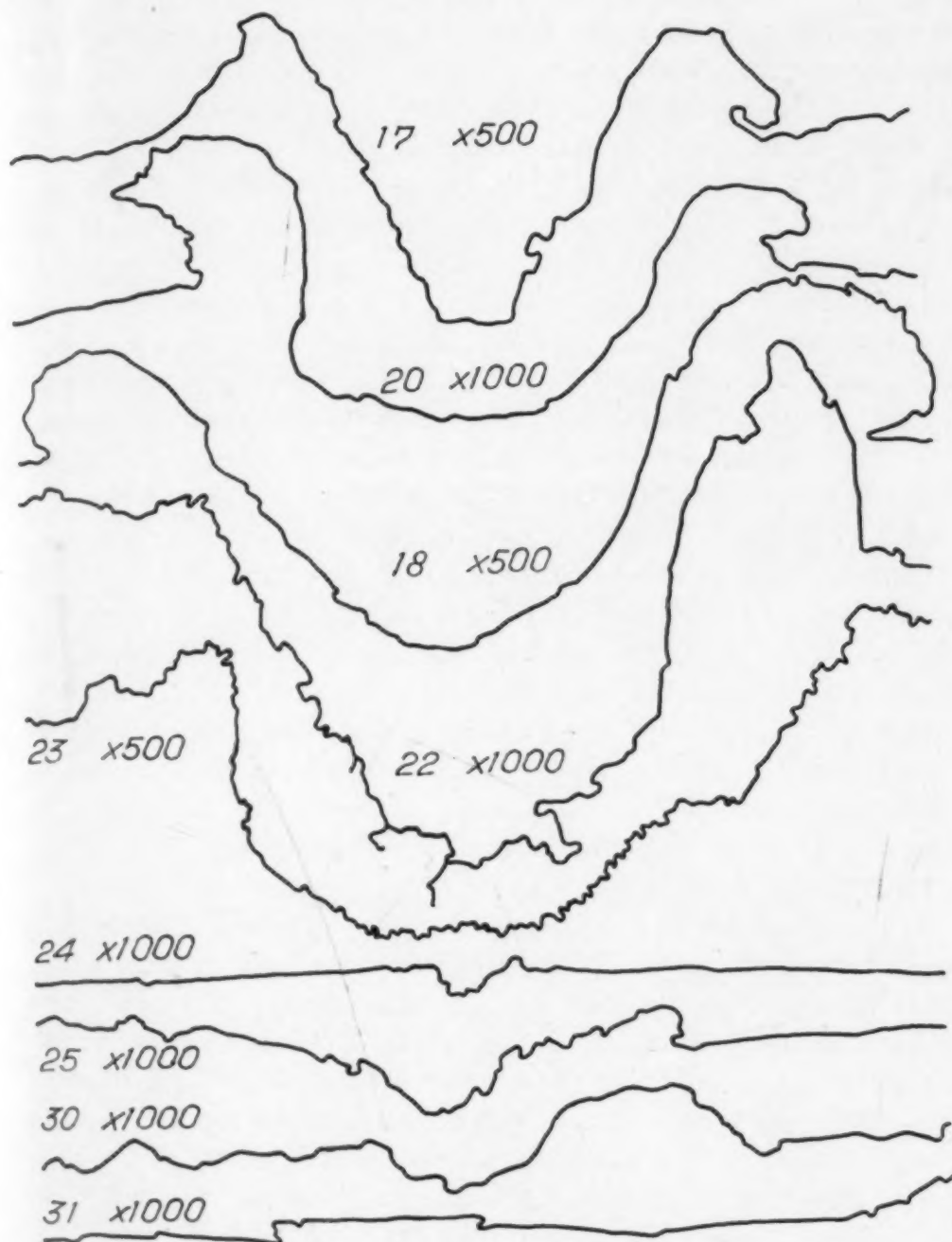


Fig. 19 (Upper)—Photomicrograph of Specimen Showing Scratch Made by 45-Degree High Speed Tool, Intended Depth—0.10 Inch, Actual Depth—0.0042 Inch, Endurance Limit—95,000 Pounds Per Square Inch, Per Cent Decrease in Endurance Limit—13.6.  $\times 250$ .

Fig. 21 (Lower)—Photomicrograph of Specimen Showing Scratch Made by 30-Degree Stellite Tool, Intended Depth—0.001 Inch, Actual Depth—0.00063 Inch, Endurance Limit—100,000 Pounds Per Square Inch, Per Cent Decrease in Endurance Limit—9.0.  $\times 1000$ .

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Lines Traced from Photomicrographs of Specimens Scratched in Varying Ways.  
Magnification Indicated on Each Curve.

Fig. No.	How Scratched	Intended Depth Inch	Actual Depth Inch	Endurance Limit Pounds Per Square Inch	Per Cent Decrease in Endurance Limit
17	45° H.S. Tool	0.002	0.0015	90,000	18.1
20	30° Stellite Tool	0.0005	0.00056	105,000	4.5
18	45° H.S. Tool	0.005	0.0019	105,000	4.5
22	30° Stellite Tool	0.002	0.0014	95,000	13.6
23	30° Stellite Tool	0.005	0.0025	95,000	13.6
24	30° Diamond Tool	0.00025	0.00012	105,000	4.5
25	30° Diamond Tool	0.0005	0.00022	95,000	13.6
30	Surface of Etched Wire.				
31	Surface of Commercial Spring Wire.				

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the point of the tool wears down to a greater extent and the sharpness of the scratch decreases. This is illustrated by several examples. In the case of the 30-degree tool the endurance limits for the 0.002-inch and the 0.005-inch scratches are the same. For the 45-degree tool the endurance limits for the 0.005-inch and 0.010-inch scratches are higher than that for the 0.002-inch scratch. In the case of the 15-degree tool the endurance limits for the 0.001-inch and 0.002-inch scratches are the same. It will be noted that scratches made by this tool lowered the endurance limit less than any of the other scratches investigated. All of the 0.0005-inch scratches made with high speed tools failed to lower the endurance limit appreciably.

The endurance limits of test specimens scratched with the Stellite tool are given in Figs. 20 to 23, inclusive. With the exception of the 0.0005-inch scratch, the results are somewhat similar to those obtained by use of high speed steel tools. For scratches over 0.001-inch deep the endurance limit is not decreased quite as much as is the case when high speed steel tools are used. This is probably due to the fact that the Stellite is more brittle than the high speed steel and the point crumbles away more rapidly in the deeper scratches. The 0.005-inch scratch made with the Stellite tool decreased the endurance limit by 5000 pounds per square inch. Apparently the greater hardness of Stellite leads to the formation of a sharper scratch at this depth.

The diamond tool scratches were apparently the sharpest of any of those investigated, since they decreased the endurance limit more than any other scratches of the same depth. They are shown in Figs. 24 to 26, inclusive. A scratch 0.0005-inch deep lowered the endurance limit to 95,000 pounds per square inch. Unfortunately, this depth was about the maximum that could be reached with the diamond tool without seriously damaging the point. This is apparent from the fact that the endurance limit of the 0.001-inch scratch is no lower than that for the 0.0005-inch scratch.

The lowest endurance limit determined on scratched specimens was that of the specimens scratched with the 30-degree high speed steel tool to a depth of 0.005-inch. This endurance limit was 90,000 pounds per square inch. The actual depth of the scratch was 0.0042 inch. The smallest radius of curvature at the bottom of the scratch was approximately 0.0014 inch. The ratio of depth to radius of curvature is exactly 3. According to the work of Griffith, as given in his Table I, the factor by which stress in the outer fibre should

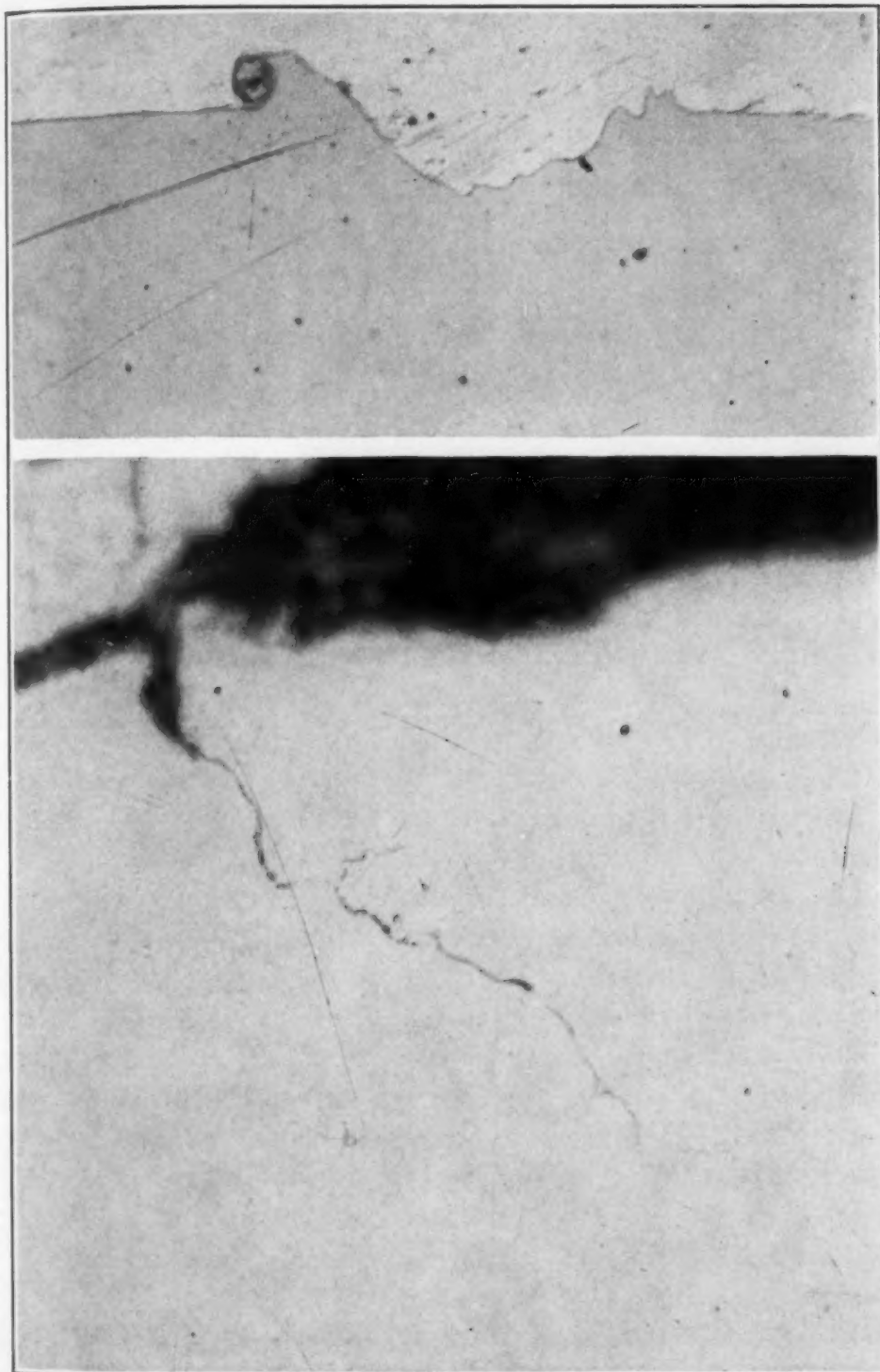


Fig. 26 (Upper)—Photomicrograph of Specimen Showing Scratch Made by 30-Degree Diamond Tool, Intended Depth—0.001 Inch, Actual Depth—0.00039 Inch, Endurance Limit—95,000 Pounds Per Square Inch, Per Cent Decrease in Endurance Limit—13.6.  $\times 1000$ .

Fig. 27 (Lower)—Photomicrograph of Surface of Defective Wire, Depth of Defect—0.003 Inch.  $\times 1000$ .

be multiplied to get the stress concentration at the bottom of the scratch is between 2.54 and 2.66. If the average of these two factors, namely, 2.60, is used, the stress concentration produced by the scratch would be 234,000 pounds per square inch. This is quite plainly a ridiculous figure, since it is much above the ultimate torsional strength of the steel. It appears, therefore, that no practical information can be obtained regarding the stress concentration in scratched wires by the use of his Table I.

As mentioned above, the lowest endurance limit obtained on a scratched test specimen was 90,000 pounds per square inch. The endurance limit of a polished test specimen was 110,000 pounds per square inch. Fatigue tests on helical springs made by F. P. Zimmerli indicated that the endurance limit of the wire was about 70,000 pounds per square inch. Figs. 27 and 28 are photomicrographs of a very poor quality spring wire which contains seams as well as scratches. It will be noted that none of the scratches were as sharp or as deep as some of those used upon test specimens in this investigation. It would appear, then, that the lower endurance limit of wire tested in the form of springs is not due entirely to the presence of surface scratches. The presence of hair-line seams in wire would be more serious than surface scratches, since the stress concentration at the inner edge of the seam could be very high.

Photomicrographs were made of the surface of the defective spring wire after it had been acid etched. A typical one is shown in Fig. 29. A comparison of these photomicrographs with those taken before acid etching showed that the etch exaggerates or emphasizes the scratches. It also tends to roughen the surface of the wire, due to a preferential attack of the steel by the etching reagent in some places. Fig. 30 shows the effect of acid etching upon the surface of the wire. The surface before acid etching is shown in Fig. 31.

#### SUGGESTIONS FOR CORRELATED INVESTIGATIONS

It is evident that in the present investigation only the surface of the problem has been scratched, in more senses than one. The endurance limits of polished spring wire test specimens have been shown to be considerably higher than those of wire specimens which have not been machined or polished. It also has been shown that scratches made upon polished test specimens lower the endurance



limit in repeated torsion appreciably. The decrease in endurance limit produced by the presence of scratches, however, did not fully

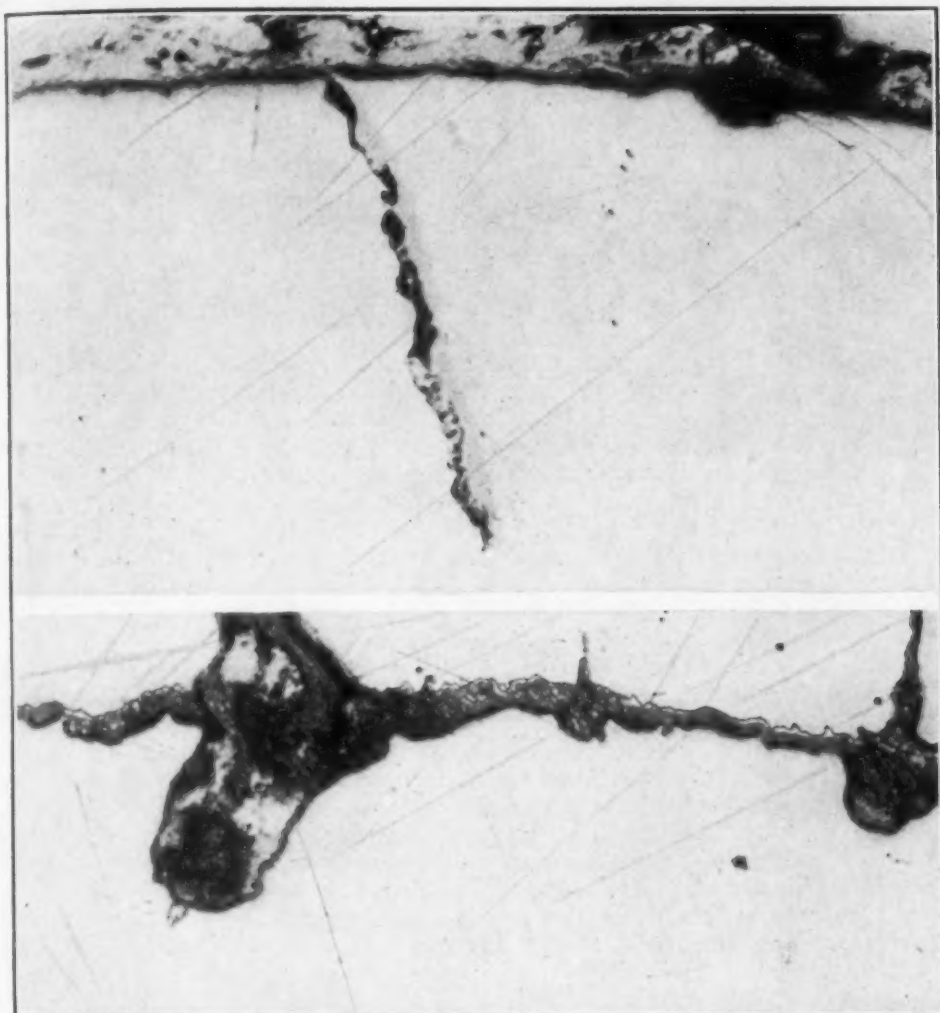


Fig. 28 (Upper)—Photomicrograph of Surface of Defective Wire, Depth of Defect—0.008 Inch.  $\times 250$ .

Fig. 29 (Lower)—Photomicrograph of Surface of Etched Defective Wire, Depth of Defect—0.0043 Inch.  $\times 250$ .

account for the difference in endurance limits of polished specimens and commercial wire in the shape of helical springs. Some English investigators suggest that this discrepancy is due to surface decarburization and oxidation. In the present investigation there was no decarburization of the wire used, so the decreases in endurance limit could not be assigned to this cause.

It is evident that the contours of surface scratches are much more important in causing a decrease in endurance limit than are the depths of the scratches. The former have a profound effect upon

stress concentration, while the latter do not affect it so much. Mechanical defects such as scratches produced during the drawing and coiling operations can never be as serious in causing spring failures as seams produced during the original processing of the wire material.

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## DISCUSSION

A. V. DE FOREST:<sup>1</sup> I would like more information on the fatigue strength of the wires with natural notches of the hair line seam type and various other kinds of seams which can occur. There are at least three different types of defects that can occur on the surface of the wire that have to be distinguished, those that were inherited from the steel as it came from the rod mill, those that came from previous drawing operations, and the final wire drawing which leaves a notch of the type we have seen on the screen; in other words, a little groove which has a radius at the bottom and which, while it is alarming to the layman who looks at it, is relatively harmless as far as their effects are concerned. We need to know more about the fatigue testing of wire and also we need additional information on identifying these various types of defects in order

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to know just what precautions are necessary, what precautions are commercial, and how to get the best spring wire at the lowest possible price.

I would like to draw attention to the great difference between the fatigue failure of cold drawn wire springs and the heat treated springs dealt with in this paper. The detriment caused by various defects is quite different in the two cases, as is also the relationship between longitudinal and transverse properties. I hope that the authors continue their work and include the music wire type of spring in their investigation of the effects of commercial imperfections in spring wire.

BRADLEY STOUGHTON:<sup>2</sup> The authors have brought out the very important point of the depth of the scratch versus the contour of the scratch, and have said that in their opinion the contour of the scratch is more important than the depth. I agree with that conclusion, but we must recognize that there is a difference of opinion on that point and others may take the ground that the depth of the scratch has some, if not a controlling interest, in the reduction of the fatigue limit. The authors and Prof. de Forest have both brought out the importance of the direction of the scratches and particularly the difference between the scratch made in the dies, a longitudinal scratch, and those made similar to it, and a scratch which may be transverse, which is inherited from the steel. The authors have also brought out the importance of a smooth tool. Perhaps many of you will remember a paper by F. T. Sisco several years ago on failures in aircraft, in which the reason for a failure defied detection until they finally found that it was due to a rough cutting tool which made a sharp scratch in what would otherwise have been a good fillet.

#### Authors' Closure

Perhaps there were a few points about this investigation that were not made clear in the presentation and for this reason the question of seams was brought up.

The intention was to first prepare as perfect a surface as possible and then to make scratches upon this surface in order to determine their effect upon the endurance limit. We tried to produce as sharp a scratch as possible, but found it was not easy to produce a deep scratch which was also sharp.

We originally planned to continue this investigation and use wire specimens that would not have a reduced diameter. In other words, we would test the full cross section of the wire. In this way, it would be possible to secure a wire that had seams and investigate the effect of seams upon the endurance limit. Unfortunately, that has not been done as yet. We hope sometime to go ahead with this and find out something about the effect of seams as A. V. de Forest suggested.

We unfortunately have not done very much work with cold drawn materials. Our interest, of course, lies largely in the heat treated spring wire, since all of our valve springs are made from this material. For this reason, we are not able to tell much about the effect of such scratches on cold drawn wire.

<sup>2</sup>Lehigh University, Bethlehem, Pa.

## METAL COLORING

By C. B. F. YOUNG

### *Abstract*

*Methods of coloring various metals and alloys by chemical and electrochemical processes are given.*

*Research has aided the metallurgist in applying hard, durable, fast colors which are in some cases resistant to corrosion.*

*Chemical solutions are given for coloring aluminum, brass, cadmium, chromium, copper, iron, magnesium, silver, tin, and zinc.*

*Electrochemical processes are given for coloring aluminum, chromium, magnesium, nickel, cobalt, and tin.*

*A very interesting black coating has been developed for magnesium and tin. This is produced in a chromic acid-trisodium phosphate solution using fifteen to thirty amperes per square foot. The process holds promise for coloring other metals but is unsuccessful when applied to iron and zinc.*

*An alloy deposit consisting of cobalt and nickel is proving to be of commercial importance as the coating produced is whiter than nickel and has better corrosive resisting properties. The color approaches that of pure silver.*

*The need for systematic research is very great. Some encouragement should be given by industrial concerns and scientific societies to those interested in this particular field. Very little has been done and much remains to be discovered. The future in this field looks very bright due to the fact that the public is demanding metallic products be finished in attractive colored designs.*

THE coloring of metals has attracted man ever since these materials have been used by the human race. Today the subject of colors is just as important, if not more so, as the attractiveness of a product depends to a great extent upon its color.

This paper deals with methods for producing a colored surface either by chemical or electrochemical processes but does not take up the subject of lacquering, painting, etc. Furthermore only those

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metals which are of commercial importance, namely, aluminum, cadmium, chromium, cobalt, copper, gold, iron, lead, magnesium, nickel, silver, tin and zinc, are discussed.

### ALUMINUM

Aluminum is a silver white metal which produces white or colorless salts. Therefore for coloring this metal it is essential to use other metals or their salts for the production of colored deposits.

A dead white coating can be produced on aluminum by boiling the metal in a solution of—

Calcium oxide, CaO	10 grams per liter
Calcium sulphate, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	10 grams per liter

A coating 0.001-inch thick is produced in ten minutes. The color tends to become grey if aluminum alloys are used. The abrasive resistance of the coating is low but it adheres well. It is possible to dye these coatings by immersing them in a suitable colored organic or inorganic solution.

Aluminum can be colored black by immersing in either of the following solutions—

Solution I	
Potassium permanganate, $\text{KMnO}_4$	10 grams per liter
Nitric acid, $\text{HNO}_3$ sp. gr. 1.35	4 cubic centimeters
Copper nitrate, $\text{Cu}(\text{NO}_3)_2$	25 grams per liter
Solution II	
Arsenous acid, $\text{As}_2\text{O}_3$	25 grams per liter
Hydrochloric acid, $\text{HCl}$ sp. gr. 1.30	50 grams per liter
Ferric sulphate, $\text{Fe}_2(\text{SO}_4)_3$	10 grams per liter

By varying the condition, it is possible to produce brown deposits on aluminum by using the above solutions.

It is possible to deposit other metals and alloys on aluminum but special care must be taken. As this field of coloring is relatively unimportant, this will be omitted.

Aluminum may be colored any hue of the rainbow by first producing an oxide coating on the surface and then allowing this to absorb and react with organic dyes, forming insoluble lakes. The colored compounds thus formed are integral parts of the metal.

Oxide coatings can be produced on aluminum by making this metal the anode in a suitable solution. There are many solutions for producing the oxide finish but three are commercially important. These are (1) the sulphate, (2) the chromic acid and (3) the oxalic acid method. The first method produces coatings which are most



suited for coloring as the film produced is practically colorless, transparent and hard. The second process produces coatings which have a slight greyish tinge and somewhat opaque. The third type produces finishes which have a slight yellow tinge and are almost transparent.

From the standpoint of coloring, the sulphate solutions are more desirable than the other two, as the chemicals used are cheap, non-poisonous, and the process is completed in about fifteen minutes. The apparatus required is rather inexpensive as no special machinery is used. The process consists of making aluminum the anode in a solution containing—

Sulphuric acid,  $H_2SO_4$  sp. gr. 1.86      100-1500 grams per liter

Glycerine or other inhibitors may be used in small amounts to prevent undue etching. The current density used should be 15-25 amperes per square foot for fifteen to twenty minutes. The solution temperature should be 65-95 degrees Fahr. The coatings thus produced vary in thickness from 0.0003 to 0.001 inch and have a hardness of approximately eight on Moh's scale of hardness.

If the oxide coating is now dipped in an aqueous solution of organic dyes, the colored chemical compound reacts with the aluminum oxide forming an insoluble lake which is precipitated throughout the coating. Acid dyes are generally used which are soluble in water. The colored materials thus formed are not absolutely stable. If exposed to sunlight, most of the materials will bleach out in twelve to eighteen months.

For this reason research has been stimulated so as to find methods of using inorganic coloring materials. The author has produced beautiful blue and blue-green colors in the oxide finishes by first soaking the anodized part in a solution containing 100 grams per liter ferric sulphate and then in a potassium ferrocyanide or ferricyanide solution containing 75 grams per liter. The blue color thus produced is fast to light. Yellow coatings have also been formed by immersing the coatings in a solution of lead acetate and then in a solution of potassium chromate. This produces the insoluble lead chromate which is an attractive yellow color.

These oxide coatings are rather porous and will absorb and/or adsorb many different types of materials. Thus, the oxide coating immersed in "aqua Dag" (colloidal carbon in water) assumes a brown black color which is permanent. The author has also found it pos-

sible to allow light-sensitive silver salts to be impregnated in the coating and on exposure to light, a dark color is produced.

Aluminum if alloyed with copper in the ratio of  $\text{Al}:\text{Cu} = 7:93$  produces a hard yellow alloy which has color similar to gold. This alloy should be used more in the field of nonferrous metallurgy.

### CADMIUM

This metal may be colored black by immersing in the following solution:

Potassium chlorate, $\text{KClO}_3$	6 grams per liter
Copper chloride, $\text{CuCl}_2$	7 grams per liter
Browns may be produced by immersing in the following solution:	
Copper Nitrate, $\text{Cu}(\text{NO}_3)_2$	30 grams per liter
Potassium permanganate, $\text{KMnO}_4$	2.5 grams per liter
Temperature	175 degrees Fahr.
Other browns may be produced by immersing in sodium thioantimonate,	
$\text{Na}_2\text{SbS}_4 \cdot 9\text{H}_2\text{O}$	30 grams per liter

### CHROMIUM

There is very little demand for coloring chromium as a metal. However, now and then it is desired to color chromium plate. This can be accomplished by several methods. A rather attractive black which has all the protective qualities of chromium is produced by immersing chromium or chromium plated objects in molten sodium cyanide at 1400 degrees Fahr. for two minutes. Remove and quench in cold water. Wash free of any adhering salts.<sup>1</sup>

It is also possible to obtain colored chromium deposits by electrolytic methods. The process consists of the ordinary bath of chromic acid but instead of using the sulphate radical, organic acid radicals are substituted such as acetate, propionate, etc. These baths give, among other deposits, deep attractive black and brown colors.

Chromium can be colored by heat treatment as it produces colored compounds. Attractive green coatings can be obtained on this material, which are close-grained, and adherent, by heating to approximately 1150 degrees Fahr. in a controlled oxidizing atmosphere. Steel wire which has been previously coated with chromium can be ingeniously colored green by this process.

### COPPER AND BRASS

These two materials are colored in the natural state, copper being a red, while brass is a dark bluish grey to a yellow red. However,

<sup>1</sup>U. S. Patent 1,937,629.

the color of brass is derived from the copper and not the zinc. Copper is rather easy to color as most of its salts are colored.

A list of formulas for coloring these two materials is given below:

### Black

Brass:		
Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$		30 grams per liter
Ammonium hydroxide. Add until precipitate of cupric hydroxide almost disappears.		
Cupric carbonate, $\text{CuCO}_3$		400 grams per liter
Ammonium hydroxide, $\text{NH}_4\text{OH}$	26 degrees	3500 cubic centimeters
Temperature		175 degrees Fahr.
Copper:		
Potassium sulphide, $\text{K}_2\text{S}$		15 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$		240 grams per liter
Arsenous acid, $\text{As}_2\text{O}_3$		30 grams per liter
Hydrochloric acid, $\text{HCl}$ sp. gr. 1.30		200 grams per liter
Sulphuric acid, sp. gr. 1.86	5 cubic	centimeters per liter
Temperature		180 degrees Fahr.

### Brown

Brass:		
Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$		60 grams per liter
Nickel sulphate, $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$		15 grams per liter
Sodium hyposulphite, $\text{NaHSO}_3$		60 grams per liter
Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$		30 grams per liter
Potassium chlorate, $\text{KClO}_3$		60 grams per liter
Barium sulphide, $\text{BaS}$		10 grams per liter
Temperature		140 degrees Fahr.
Copper:		
Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$		30 grams per liter
Potassium chlorate, $\text{KClO}_3$		10 grams per liter
Temperature		175 degrees Fahr.
Ammonium sulphide $(\text{NH}_4)_2\text{S}$		conc.
Copper acetate $\text{Cu}(\text{Ac})_2$		30 grams per liter
Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$		24 grams per liter
Barium sulphide, $\text{BaS}$		24 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$		240 grams per liter
Temperature		110 degrees Fahr.

### Blue

Brass:		
Arsenous acid, $\text{As}_2\text{O}_3$		115 grams per liter
Hydrochloric acid, $\text{HCl}$	750 cubic	centimeters per liter
Water	200 cubic	centimeters per liter
Copper:		
Potassium chlorate, $\text{KClO}_3$		100 grams per liter
Ammonium nitrate, $\text{NH}_4\text{NO}_3$		100 grams per liter
Copper nitrate, $\text{Cu}(\text{NO}_3)_2$		2 grams per liter



**Green****Brass:**

Copper nitrate, $\text{Cu}(\text{NO}_3)_2$	30 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$	30 grams per liter
Copper nitrate, $\text{Cu}(\text{NO}_3)_2$	30 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$	10 grams per liter
Acetic acid conc., $\text{HAc}$	60 cubic centimeters per liter

**Copper:**

Copper nitrate, $\text{Cu}(\text{NO}_3)_2$	30 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$	30 grams per liter
Copper nitrate, $\text{Cu}(\text{NO}_3)_2$	40 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$	40 grams per liter
Calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	40 grams per liter

There are many other formulas for coloring brass and copper but the above will serve as a start for those desiring information in this field.

The alloys of copper are also very important as nearly all of them are colored. Important systems are copper-zinc, copper-tin, and copper-cadmium.

Thus an alloy containing 95 per cent copper and 5 per cent tin is a golden yellow. An alloy containing 65 per cent copper and 35 per cent zinc is a red yellow, etc. The designer can obtain many colors by using alloys of copper with other metals.

**Gold**

This material is never colored as a base metal but many times it is used to color other metals or alloys. Yellow gold may be plated from the bath below:

Metallic gold as cyanide	5 pennyweight per gallon
Potassium cyanide, KCN	2 ounces per gallon
Sodium phosphate, $\text{Na}_3\text{PO}_4$	5 ounces per gallon
Temperature	175 degrees Fahr.
Current density	2-10 amperes per square foot
For plating green gold:	
Metallic gold as chloride	5 pennyweight per gallon
Metallic silver as chloride	0.25 pennyweight per gallon
Potassium cyanide, KCN	4 ounces per gallon
Temperature	175 degrees Fahr.
Current density	2-10 amperes per square foot
For plating white gold:	
Gold as cyanide	5 pennyweight per gallon
Nickel as cyanide	12 pennyweight per gallon
Potassium cyanide, KCN	4 ounces per gallon

**IRON AND STEEL**

These materials can be colored by heat treating, chemical treat-

ing, and electroplating. If steel is heated in the open air the following colors result at the designated temperatures:

	Degrees Fahr.
Pale yellow	420
Straw yellow	440
Brown	490
Purple	536
Pale blue	570
Dark blue	600

Salt baths are used instead of air so that the temperature is controlled easier, and also the color can be made somewhat deeper. A representative bath contains equal parts of sodium and potassium nitrate with manganese dioxide added in a ratio of one part oxide to fifty parts of the nitrate mixture. The temperature of this bath is 600-650 degrees Fahr.

Chemical coloring of iron and steel parts is gaining ground. The coatings produced not only are attractive but also serve as protection against corrosion. Black colors can be produced by immersing in the following solutions for twenty to thirty minutes:

I	
Sodium hydroxide, NaOH	400 grams per liter
Potassium nitrate, KNO <sub>3</sub>	10 grams per liter
Sodium nitrate, NaNO <sub>3</sub>	10 grams per liter
Temperature	250 degrees Fahr.
II	
Copper sulphate, CuSO <sub>4</sub> · 5H <sub>2</sub> O	150 grams per liter
Nitric acid, HNO <sub>3</sub> sp. gr. 1.35	300 cubic centimeters per liter
Ethyl alcohol, C <sub>2</sub> H <sub>5</sub> OH	600 cubic centimeters per liter

The parts to be colored are cleaned, immersed and then allowed to dry in the air. When dry, they are rubbed with cloths containing oil which darkens the color.

Blue colors are produced by immersing in the following:	
Sodium hyposulphite, NaHSO <sub>2</sub>	60 grams per liter
Lead acetate, Pb(Ac) <sub>2</sub>	15 grams per liter
This solution is used at 212 degrees Fahr.	
For coloring cast iron blue use the following:	
Lead nitrate, Pb(NO <sub>3</sub> ) <sub>2</sub>	7.5 grams per liter
Ferric nitrate, Fe(NO <sub>3</sub> ) <sub>3</sub>	3.0 grams per liter
Sodium hyposulphite, NaHSO <sub>2</sub>	30.0 grams per liter

Sodium nitrite is also finding application as a blackening agent for steel. This material can be used in aqueous solutions to produce attractive black finishes on either iron or steel. There are many other formulas which can be used to color these materials but the baths are so numerous it would be impossible to list them in a paper as short as this.

Iron and steel may be colored red by depositing a coating of copper on the surface, yellow by depositing brass, white by depositing nickel or chromium and black by depositing black nickel on the objects. The formulas for these baths and their conditions will not be discussed here as this information is available elsewhere. However, it might be pointed out that copper or brass can be deposited on iron and steel and the coatings colored by using the methods prescribed under "Copper and Brass."

#### LEAD

This material forms some colored salts but as they all have a tendency to crumble, very little importance is placed on this type coloring. The best way in which to color lead or lead alloys is by the electrodeposition of other metals and alloys on its surface.

#### MAGNESIUM

Ordinarily this metal would be passed by with only a word as to its possibilities. However, the author has directed some research in the past few months which produced some very interesting results. It was found that if magnesium is made the anode in a solution containing various amounts of chromic acid and trisodium phosphate, and a current of 15-30 amperes passed through, a very attractive black coating was produced. A representative bath contains:

Chromic acid, $\text{CrO}_3$	100-200 grams per liter
Trisodium phosphate, $\text{Na}_3\text{PO}_4$	100-250 grams per liter
pH	approximately 3
Temperature	75-200 degrees Fahr.

The coatings produced were grey to jet black in color and rather hard. Fig. 1 shows a magnesium strip which has been treated by this method. On analyzing the deposit under a microscope, it was found that certain areas did not take the black coating. Fig. 2 shows a photomicrograph of the black deposit with certain areas failing to be covered. The peculiar construction of the white areas was rather interesting as all seemed to be long and narrow. On analyzing a part of the same piece which was untreated, certain areas seemed to be differentiated from the rest. These also were rather long and narrow. Fig. 3 shows a photomicrograph of such areas. Note the circled area and the long slender body marked "ABC." This material is undoubtedly a eutectic structure and does not behave electro-



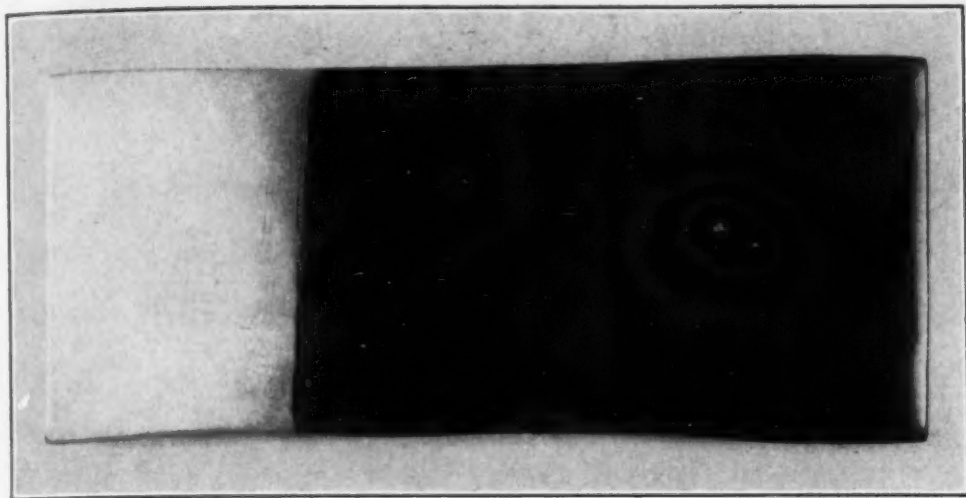


Fig. 1—Ebony Black Coating on Magnesium.

chemically as does magnesium. It undoubtedly is possible by research to obtain a coating which will cover the whole area. It was also found that this method colors tin, but does not color zinc, brass, and iron. Under certain conditions the black coating was obtained by simply immersing the magnesium or tin in the solution. More research is being conducted in this field. The author wishes to acknowledge the work of Albert Pflomm in this connection.

Buzzard and Wilson of the Bureau of Standards have been researching in this particular field and found that the following solution gives grey to black deposits also:

The solution contains:	
Sodium dichromate, $\text{Na}_2\text{Cr}_2\text{O}_7$	10 per cent
Sodium hydrogen phosphate, $\text{NaH}_2\text{PO}_4$	2-5 per cent
Anode, magnesium	
Steel cathode	
Current density, 5-10 amperes per square foot of anode area	
Temperature	50 degrees Cent.
pH	4-4.8

In order to keep the pH constant, phosphoric acid is added to the bath when necessary.

#### NICKEL AND COBALT

Nickel and cobalt finishes are not used very often for securing color coatings. However, some baths are used. One consists of treating the nickel or cobalt in the following solution:

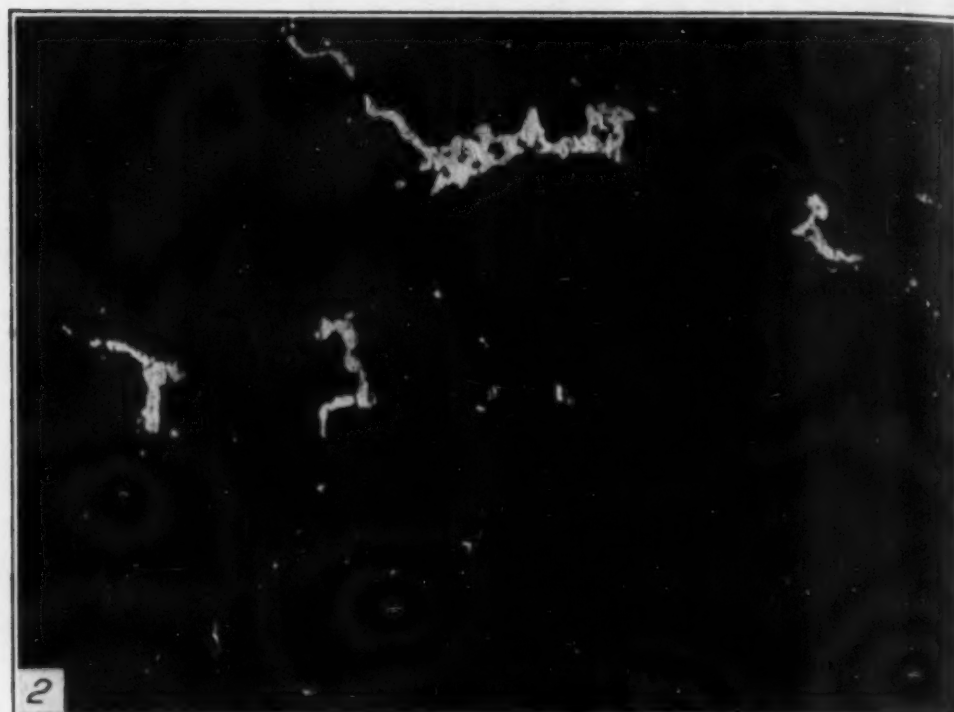


Fig. 2—Black Coating on Magnesium.  $\times 150$ .  
Fig. 3—Untreated Magnesium Base Metal Showing Eutectic Areas.  $\times 150$ .

Ammonium chloride, $\text{NH}_4\text{Cl}$	50 grams per liter
Ammonium sulphide, $(\text{NH}_4)_2\text{S}$	concentrated

Black or grey tints are obtained.

One of the best known methods of coloring nickel black is by the electrolysis of a bath containing:

Nickel ammonium sulphate, $\text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	8 ounces per gallon
Zinc sulphate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	2 ounces per gallon
Sodium sulphocyanide, $\text{Na CNS}$	2 ounces per gallon
pH	5.3-6
Temperature	80 degrees Fahr.
Current density	2 amperes per square foot, producing 1-1.5 volts

A rather interesting alloy can be deposited using nickel and cobalt. This process is in operation in several plants of the New York area. Young and Gould<sup>2</sup> used a solution containing:

Nickel sulphate, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$	195 grams per liter
Cobalt sulphate, $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$	35 grams per liter
Sodium chloride, $\text{NaCl}$	15 grams per liter
Boric acid, $\text{H}_3\text{BO}_3$	25 grams per liter

The alloy produced approaches silver in whiteness and is more corrosive resistant than either cobalt or nickel.

Electrodeposits containing nickel and sulphur were studied by W. T. Young and H. Kersten.<sup>3</sup> They used a solution containing:

Nickel ammonium sulphate, $\text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$	100 grams per liter
Sodium thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$	10 grams per liter
Sodium citrate, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 5\text{H}_2\text{O}$	15 grams per liter
pH	6.4
Current density	2-20 amperes per square foot

Greenish coatings consisting principally of  $\text{Ni}_3\text{S}_2$  were formed.

## SILVER

Silver is one of the whitest metals known, being the standard for measuring reflectivity. The metal is practically inactive as far as oxygen is concerned, but reacts readily with sulphides, forming brown or black deposits. The following solution may be used:

Ammonium sulphide, $(\text{NH}_4)_2\text{S}$	10 grams per liter
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By varying the concentration of the above material and the time of immersion, various shades of brown can also be obtained.

<sup>2</sup>"Nickel Cobalt Alloy Plating" by Young and Gould, *Transactions, American Electrochemical Society*, Vol. 69, 1936, p. 585-597.

<sup>3</sup>*Transactions, American Electrochemical Society*, Preprint 71-4, p. 33-37.



## TIN

This element is of interest due to its large usage as a protective coating for steel. If tinned steel can be made attractive, there is no reason for the process not being commercially successful. Research is beginning to produce results in this field. As stated before, the author found that a chromic acid solution produced a deep black on tin provided certain conditions were used. This coating was very adherent and withstood severe binding and scratching. More research is being conducted on this interesting coating.

R. Kerr and D. J. MacNaughton also produced black coatings on tin by anodizing the material in a solution containing:

Sodium phosphate, $\text{Na}_2\text{HPO}_4$	200 grams per liter
Phosphoric acid, $\text{H}_3\text{PO}_4$ Den. 1.75	20 cubic centimeters per liter
Current density	12-48 amperes per square foot
Temperature	60-90 degrees Cent.

A six-minute treatment produced a blue black coating 0.0001-inch thick.

## ZINC AND ZINC-BASE DIE CASTING

An ebony black deposit is produced by immersing the zinc in the following:

Copper nitrate, $\text{Cu}(\text{NO}_3)_2$	7.5 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$	10 grams per liter
Cupric chloride, $\text{CuCl}_2$	7.5 grams per liter
Hydrochloric acid, $\text{HCl}$ sp. gr. 1.3	300 cubic centimeters per gallon
Temperature	100 degrees Fahr.
Allow to dry and heat to	160 degrees Fahr.

The solution below produces black on zinc:

Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	150 grams per liter
Potassium chlorate, $\text{KClO}_3$	80 grams per liter

An adherent dark brown can be produced by permitting the alloy to be electrodes in a solution containing 250 grams per liter of chromic acid ( $\text{CrO}_3$ ). A low voltage alternating current is passed through the solution and the color develops in two to three minutes.

A rich brown can be obtained by immersing the material in the solution given below:

Sodium thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3$	124 grams per liter
Lead acetate, $\text{Pb}(\text{Ac})_2$	38 grams per liter

A steel blue may be produced by immersing the metal in a solution containing:

Cobalt chloride, $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$	60 grams per liter
Ammonium chloride, $\text{NH}_4\text{Cl}$	60 grams per liter

If nickel ammonium sulphate is substituted for the cobalt salt, a bright purple coating is obtained. The latter solution should be used at 140 degrees Fahr.

Yellow, brown, crimson, blue, purple, and light green may be produced in succession on zinc by using the following solution:

Copper tartrate, $\text{CuC}_4\text{H}_4\text{O}_6 \cdot 3\text{H}_2\text{O}$	150 grams per liter
Sodium hydroxide, $\text{NaOH}$	175 grams per liter
Temperature	90-100 degrees Fahr.

A similar solution also produces iridescent sequences of colors consisting of:

Copper sulphate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	50 grams per liter
Potassium acid tartrate, $\text{KHC}_4\text{H}_4\text{O}_6$	50 grams per liter
Sodium carbonate, $\text{Na}_2\text{CO}_3$	125 grams per liter

An attempt has been made in this paper to focus attention on the processes now used in the coloring of metals. Research has been lacking in most cases and in others no attempts have been made for systematic studies. It is the hope of the author that some definite steps may soon be started which will lead to a well organized plan for research in this very interesting field.

## DISCUSSION

**Written Discussion:** By Adolph Bregman, managing editor, *Metal Industry*, New York City.

The author has presented a most interesting paper on a subject which is of growing importance to the metal products manufacturing industries. Progressive manufacturers are awake to the need for style, finish and attractiveness in order to sell to the general public, and the usefulness of color in improving appearance. The author's brief summary of present methods of coloring is useful. A complete list of formulae, etc., would necessitate a large volume, but his summary indicates well the scope and variety of this type of work.

Under the subheading Chromium on page 1022, in the first paragraph, it is stated: "A rather attractive black\*\*\*\*\* is produced by immersing chromium or chromium plated objects in molten sodium cyanide, etc." This is probably a slip of the pen as I know of no solid chromium objects unless the author means high chromium steel alloys; in which case I wonder if the sodium cyanide treatment has the same effect as it has on the pure chromium surfaces of chromium plated objects.

In the last paragraph on page 1022 under the subheading Copper and Brass, the author mentions that "Brass is a dark bluish grey to a yellow red." This is true, of course, but dark bluish grey brass is of no commercial importance, this color existing only with very high zinc content and mixtures of this type have such poor properties as to make them unfit for commercial use.

On page 1026 line 1, the author states that "Iron and steel may be colored red by depositing a coating of copper on the surface, yellow by depositing brass, etc." This to my mind is not "metal coloring." It is simply electroplating with metals of a different natural color. The term "metal coloring" is, as generally understood, restricted to the change of the natural color by chemical or electro-chemical means. The same applies to the author's statement in the following paragraph that "The best way in which to color lead or lead alloys is by the electrodeposition of other metals and alloys on its surface."

The author's discussion of a new black coating on magnesium and tin is extremely interesting and merits further work. I heartily second the author's hope that some definite steps may soon be taken which will lead to research in the field of metal coloring which is, in my opinion, only in its infancy.

#### Oral Discussion

CLAYTON H. WHITE:<sup>1</sup> I would like to ask the author one question concerning the coloring of aluminum. I believe one of the chief advantages of the dyed aluminum coatings is that they are not brittle, and aluminum in the form of sheet can be bent without the coating flaking or breaking off readily. I would like to ask if, in the case of the inorganic coloring matter where, in all probability, the coloring material is in a crystalline form, whether these coatings also are not brittle and not subject to wear.

Another question has occurred to me on this subject of aluminum coating with ferrocyanide, as to whether the author has used both of the salts in the same solution or whether the material has been treated with ferric salt and then with ferrocyanide separately?

BRUCE W. GONSER:<sup>2</sup> Metal coloring is certainly a coming field. However, most of the work in the past and most articles on the subject have dealt with surface coloration only. I wish to point out that natural coloration throughout the metal product, as differentiated from surface coloration, should be included in a discussion of this type. Many metal products can be colored naturally by choice of a suitable alloy composition. Of course, there is not a great diversity of colors in the common metals and alloys but surprising colors are sometimes developed in making alloys, and research for this purpose might develop some interesting combinations. With copper and antimony, for example, the color of copper soon turns to a white or colorless alloy as antimony is added, then with about 50 per cent of each a deep purple color develops as the compound  $Cu_2Sb$  is formed. There are various other examples of unexpected color development, such as the pinkish coloration obtained with certain cadmium-silver alloys, although each of the constituents is white.

With a product that is uniformly colored less care need be taken of the

<sup>1</sup>Farrel-Birmingham Co., Ansonia, Conn.

<sup>2</sup>Metallurgist, Battelle Memorial Institute, Columbus, Ohio.



surface. Scratches show but little. If the surface gets dull the original color is restored by polishing, grinding or liberal use of sandpaper. Many of these colored intermetallic compounds, such as  $\text{Cu}_2\text{Sb}$ , are very brittle and for some purposes would be worthless, but for fairly large cast sections which have no thin fins even such poor physical properties might be subordinate to the advantage of an attractive color.

One of the new branches of this field is that of using color in powdered metallurgy, or where products are made from compressed metal powders. Since several constituent metal powders may be used, some of which may be colored, it is possible to obtain various color effects that are homogeneous throughout the product, as previously described, rather than being merely on the surface. As examples, one may use copper or brass powders to get a desired red or yellow and the copper-antimony intermetallic compound  $\text{Cu}_2\text{Sb}$ , for a purple shade. The possibilities in this field for actually getting color designs that will not wear off and without interfering too much with other desired physical properties are really promising.

K. R. VAN HORN:<sup>3</sup> Perhaps the most striking illustration of coloring by alloying is the copper-zinc system, where the colors may vary from the yellow and red of the high-copper alloys to the blue and gray of the zinc-rich alloys. This illustration was described by the author in the manuscript.

There are two items regarding the coloring of aluminum which perhaps the author may care to amplify. He mentions that the anodic treatment of aluminum requires 15 to 20 minutes, while in commercial practice the usual period is about 30 minutes. When the anodic film is to be pigmented, an additional 10 or 15 minutes is necessary, so that the entire process of anodizing and coloring consumes about 50 minutes.

The author states that a blue color is produced by ferric sulphate and either potassium ferro- or ferricyanide. According to the chemistry of reactions, a ferric salt and ferrocyanide produce Prussian blue. However, a ferric salt and a ferricyanide will give no color. The reaction of a ferrous salt and ferricyanide produces a blue (Turnbull's) color. It is possible that the author meant to state that a ferric salt and potassium ferrocyanide produce the blue color in question. However, this blue (Prussian) is not permanent as the color fades if exposed to sunlight for a period of two months.

The two photomicrographs of the coloring of magnesium sheet are interesting. The author attributes the small discontinuous white spots in the coating to the presence of a eutectic. These photographs are undoubtedly of pure magnesium or of magnesium alloy sheet. The only two magnesium alloys that are fabricated in sheet form are known as AM3S and AM57S. Neither of these alloys nor pure magnesium contain a eutectic and consequently I would suggest that dross films are possibly the cause of the imperfections or discontinuities in the black coating on magnesium.

#### Author's Closure

I might say that during the last two or three months there has been a new process put on the market by the United Chromium Company called electro color, a method of obtaining any color you like. The material is made up in

<sup>3</sup>Aluminum Company of America, Cleveland, Ohio.

the cathode containing organic copper compounds. I think copper lactate is a good example. By varying the time and the consequent current, they can get almost any color of the rainbow. I have some samples here that were sent me just before I came to this meeting. They are attractive, but must be lacquered to protect the coloring, with any type of clear lacquer. They include greens, red, blues, darker greens, rose colors, etc.

In answer to C. H. White, allow me to say that the only way the colored coating can be broken is to break the metal under it. The inorganic coating will hold just as well as the dyed coatings. The explanation seems to be that the finely divided coloring material is precipitated through the interstices of the aluminum oxide and this film has to be broken in order to break the colored coating. In regard to Mr. White's second question, separate solutions are used for each salt.

I agree with Dr. Gonser concerning the use of alloys for coloring. In the paper I did mention that alloying was a method of obtaining colors and as an example the zinc-copper series was used. I did not go into great detail at this point as the information would have been rather voluminous. I also agree with Dr. Gonser concerning the use of metallic powders for obtaining colored objects. This method bids to become one of the most important means of obtaining colors in metallic objects.

In reply to Dr. Van Horn let me say that fifteen to twenty-five minutes is ample time in which to produce an oxide coating on aluminum. In my research the above period has produced coatings which work well. Several hundred pieces of aluminum have been colored by the author, using oxide coatings which were produced in fifteen minutes. I agree with Dr. Van Horn concerning the chemistry of the ferro- and ferricyanides. In my research I have found that a coating dipped in a solution containing a ferricyanide will produce a color if the coating is now dipped into a ferric salt solution. This is due to the fact that some aluminum is present which reduces, partially, the ferric solution to ferrous solution. These materials unite to give Turnbull's blue. In order to obtain these colors, it was necessary to treat the coatings for a few minutes at an elevated temperature.

I have some aluminum sheets which were colored blue about three years ago. So far, they have not faded. During this time they were on my desk where they were exposed to sunlight. I have not, however, made a direct exposure test on these pieces. Dr. Van Horn seems to have a very logical suggestion concerning the imperfections in the magnesium coatings.

In regard to pure chromium objects, I agree with Mr. Bregman, but this material comes from a Du Pont patent and is stated in this manner. For this reason "chromium or chromium plated" was used.

In reply to the criticism that dark bluish grey brass is not used commercially due to its poor characteristics I also agree. However, this alloy could be used as a coating for iron or steel, producing thereby a color which would have strength.

Mr. Bregman objects to my calling electroplating a method of metal coloring. To me, the definition of metal coloring includes the electrodeposition of metals, and therefore it is included here.

## A NEW APPLICATION FOR THE SHORT-TIME HIGH TEMPERATURE TENSILE TEST

BY C. L. CLARK, A. E. WHITE AND G. J. GUARNIERI

### *Abstract*

*This paper presents results obtained from short-time high temperature tensile tests on five steels over a range of temperatures. The rate of load application, and thus the rate of deformation as well as the time for fracture, was varied and the resulting tensile strength values were correlated with the speed of testing.*

*On this basis an apparently critical temperature was found to exist which differed for each of the analyses considered. Furthermore, the magnitude of this temperature appears to give an indication of the creep characteristics at temperatures in its vicinity.*

*This method may serve therefore as a type of an acceptance test in indicating whether or not steels of the same general type and composition possess the same creep strength.*

THE art of high temperature testing has progressed rapidly during the past few years and, as is often the case, there is the possibility that some of the earlier tests which have been largely discarded might again prove to be of value, at least in a supplementary capacity, when viewed with our present knowledge. This is believed to be the present status of the short-time high temperature tensile test.

It is true that the majority of laboratories interested in high temperature investigations do conduct tensile tests over a rather wide temperature range. In general, however, designers base their calculations on the creep characteristics and use the tensile properties mainly as an indication of the metal's ductility over the proposed operating temperature range. One oil company has, however, used the tensile test at 1600 degrees Fahr. (870 degrees Cent.) in the selection of cast materials with great success.

While the creep characteristics are now employed by many engineers for design purposes, they possess two inherent disadvantages.

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. Of the authors, C. L. Clark and A. E. White are Research Engineer and Director, respectively, Department of Engineering Research, University of Michigan, and G. J. Guarnieri is graduate student, University of Michigan. Manuscript received June 25, 1937.



The first of these is the extended time period required for their determination and the second is the fact that different heats with the same chemical composition and heat treatment do not necessarily possess the same creep properties. It is possible, therefore, that even though considerable care and effort are spent in determining the creep characteristics of a given analysis, the material finally placed in service, if from a different heat, may have appreciably different load-carrying ability.

Realizing the shortcomings of the present-day creep test, it was believed advisable to further investigate the short-time high temperature tensile test in order to determine if it might be of some value in indicating the creep characteristics and in serving as an acceptance test. A great need exists for a test of this latter type for it would be practically impossible to conduct the present standard creep test on every heat of steel.

#### GENERAL CONSIDERATIONS

It is now generally agreed that creep is a resultant of two factors: strain-hardening and recrystallization. At the lower temperatures strain-hardening predominates while at the more elevated ones the rate of recrystallization is the governing factor. On this basis there must exist for each analysis a critical temperature which divides these two fields. The location of this temperature undoubtedly depends upon many factors among which are chemical composition and the past history of the particular heat of steel. It is further believed that for a given series of steels at a given test temperature, the one having the highest critical temperature will also possess the greatest creep resistance, provided the test temperature is not too far above or below this critical temperature.

The question now arises as to whether or not the position of this temperature can be determined from short-time tensile tests. The authors believe it can, provided one additional factor, which is the rate of loading, is taken into consideration. It is true that in the present A.S.T.M. Tentative Standard covering this test (E21-34T) a rate of loading of 0.22 to 0.28 inches per minute is specified. If this rate of loading be employed at temperatures below 1200 degrees Fahr. (650 degrees Cent.), however, considerable care must be exercised to prevent an undue rise in the temperature of the specimen during test.

## PROCEDURE

The method employed in this investigation consisted of conducting a series of tensile tests on each analysis at each temperature under varying rates of loading. The tensile strength value, the time required for fracture, and the rate of deformation were recorded and it was found that approximately straight-line relationships were obtained when either the tensile strength and time for fracture or the tensile strength and rate of deformation were plotted to logarithmic coordinates.

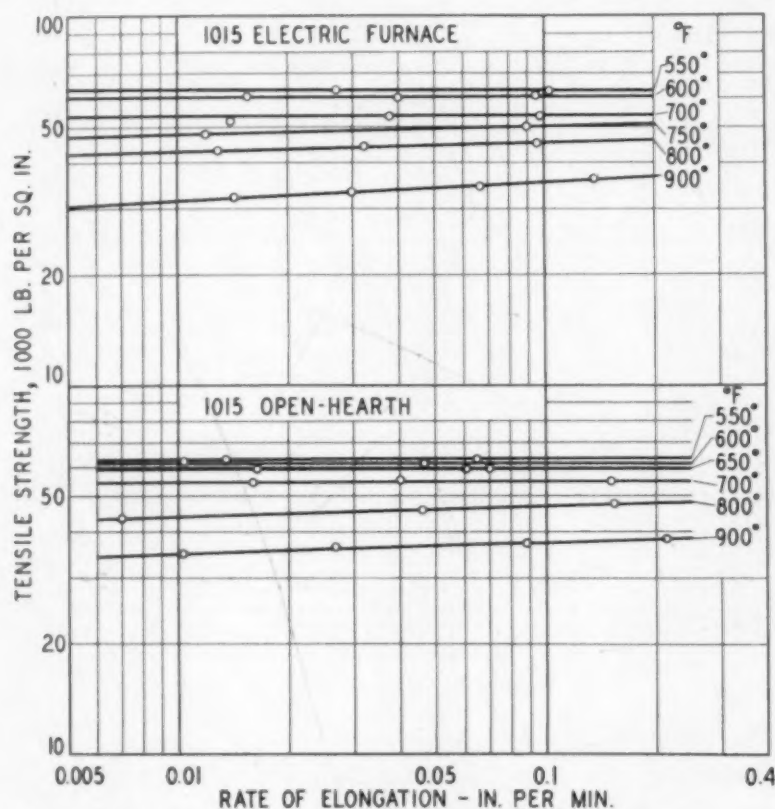


Fig. 1—Tensile Strength Versus Rate of Elongation.

Results obtained over a range of temperatures indicated that while the straight-line relationship continued to hold, the slope of the lines depended on the temperature. By plotting to semi-logarithmic co-ordinates, the temperature versus the tensile strength for a given rate of deformation or the tensile strength for a given fracture time, sets of lines with different slopes were obtained all of which, for each analysis, intersected at a common point. This point of intersection is believed to indicate a critical temperature for each

analysis. Attempts were then made to correlate this temperature with the known creep results at a common temperature.

Standard 0.505-inch diameter specimens were employed and a hydraulic tensile machine was used in all of the tests. The rate of elongation during the test was obtained by measuring, with an Ames dial, the speed of the movable head of the machine. These dial read-

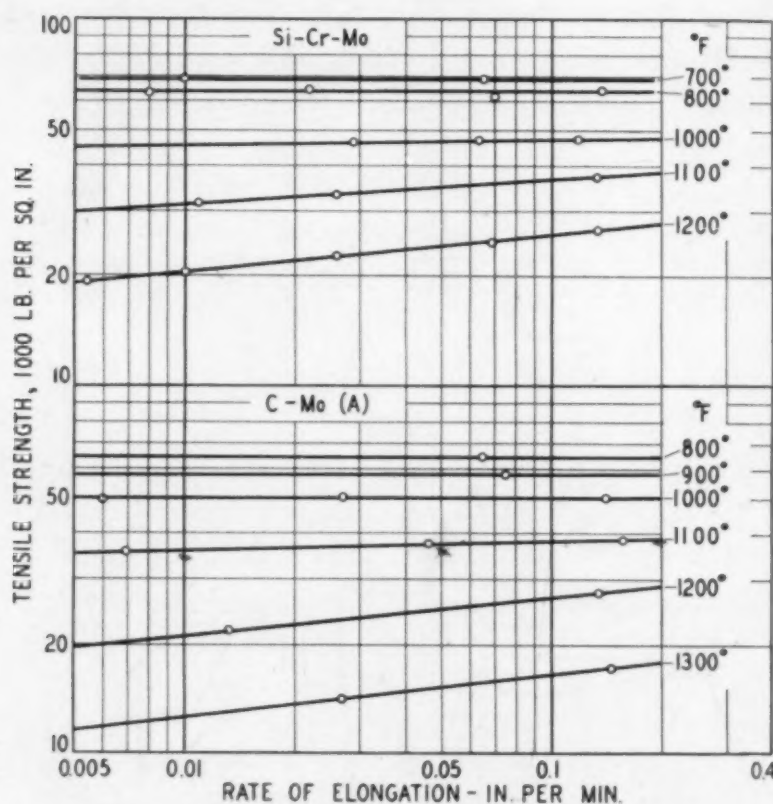


Fig. 2—Tensile Strength Versus Rate of Elongation.

ings were then plotted against time and the slope of the resulting line taken as the rate of deformation. The sensitivity of the extensometer was 0.0002 inches per inch. It is recognized that this method is not overly sensitive but, as will be pointed out later, the results thus obtained are in good agreement with those based on fracture time.

This test method should not be confused with that described by two of the authors<sup>1</sup> in a paper before this Society last year. In the previous method a fixed load is applied and the time for fracture recorded. In the present one the load is, of course, changing con-

<sup>1</sup>A. E. White, C. L. Clark and R. L. Wilson, "The Fracture of Carbon Steels at Elevated Temperatures," *TRANSACTIONS, American Society for Metals*, Vol. 25, 1937, p. 863.



tinuously during the test. Each method possesses its own advantages and neither is intended to replace the other.

### STEELS INVESTIGATED

The steels necessary for this investigation were secured from The Timken Steel and Tube Division of The Timken Roller Bearing Company. They were all furnished in the form of hot-rolled, annealed, 1-inch round bars. Their composition, hardness and grain size were as follows:

Designation	Chemical Composition					Brinell Hardness	Grain Size
	C	Mn	Si	Cr	Mo		
1015 (EI)	0.15	0.50	0.23	...	...	134	4-5
1015 (OH)	0.15	0.46	0.26	...	...	111	5-6
Si-Cr-Mo	0.11	0.41	0.78	2.50	0.50	143	7-8
CMo (A)	0.16	0.47	0.23	...	0.42	126	4-5
CMo (B)	0.17	0.52	0.16	...	0.54	...	..

All of these steels were annealed from the same temperature, 1550 degrees Fahr. (845 degrees Cent.). The two carbon analyses differ only in that one was made in an electric-arc furnace and the other in an open-hearth. The two carbon-molybdenum steels differ mainly in that they are from different heats.

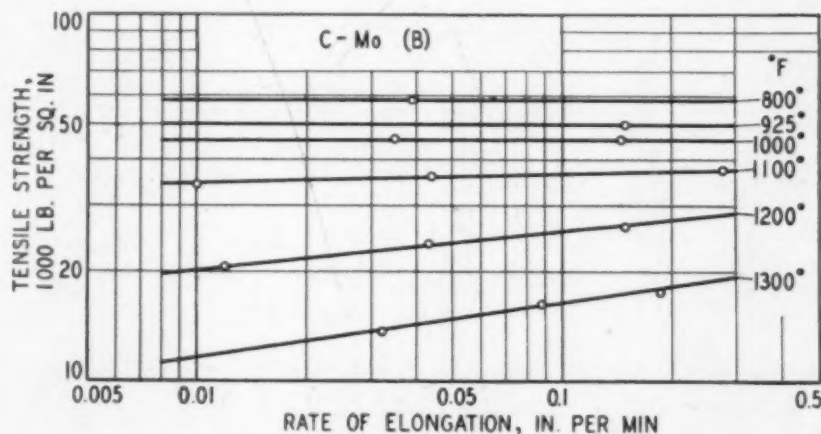


Fig. 3—Tensile Strength Versus Rate of Elongation.

### RESULTS

Each of the five analyses were subjected to tensile tests, under varying rates of loading, at several temperatures. These temperatures were chosen so that they would lie both above and below the apparently critical temperature.

### *Tensile Strength Versus Rate of Elongation*

The relationship obtained between rate of loading and tensile strength, when plotted to logarithmic co-ordinates, is shown in Figs. 1, 2 and 3. It will be noted that in all cases approximately straight-lines are obtained and that the slope of these lines depends upon the temperature and the composition.

The interesting fact is that in each case the rate of elongation, at least over the range considered, is without influence on the resulting tensile strength value at the lower temperature. This implies that at each of these lower temperatures the same tensile strength value will result regardless of whether an elongation rate of 0.01 or 0.15 inches per minute is used.

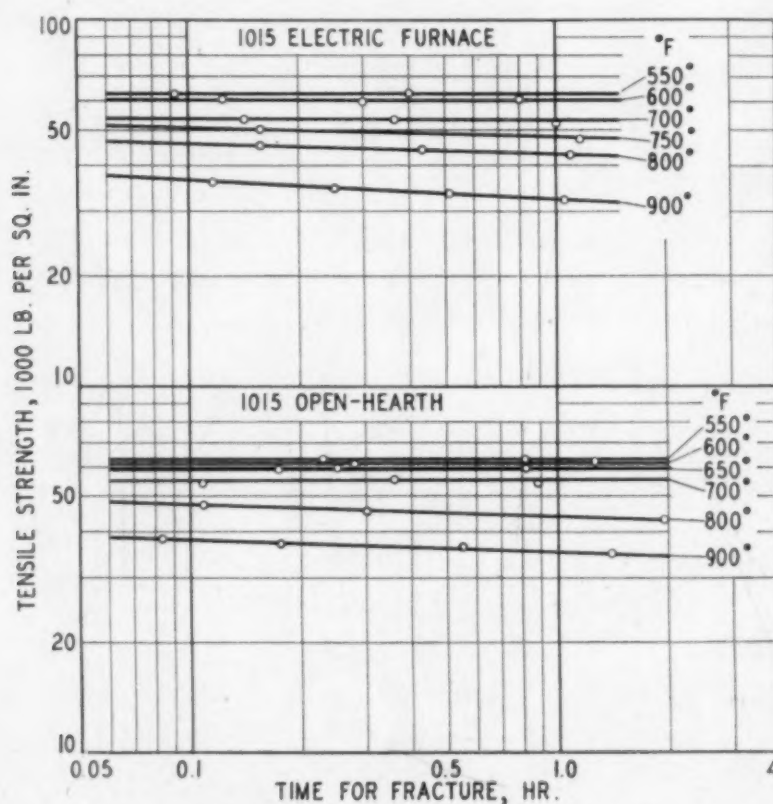


Fig. 4—Tensile Strength Versus Time for Fracture.

At the higher temperatures the tensile strength value increases as the rate of elongation is increased. This condition is as expected for the creep test may be considered as a very prolonged tensile test and it is known that it results in minimum values.

The temperature required for the logarithmic lines to assume a definite slope value varies with the different analyses. For example,

with the open-hearth carbon steel this break occurs between 600 and 700 degrees Fahr. (315-370 degrees Cent.) while with the CMo (A) steel, the corresponding temperature is close to 1000 degrees Fahr. (540 degrees Cent.).

### *Tensile Strength Versus Time for Fracture*

The same results are plotted to logarithmic co-ordinates, except on the basis of fracture time rather than rate of elongation, in Figs. 4, 5 and 6. Again it will be noted that an approximately straight-

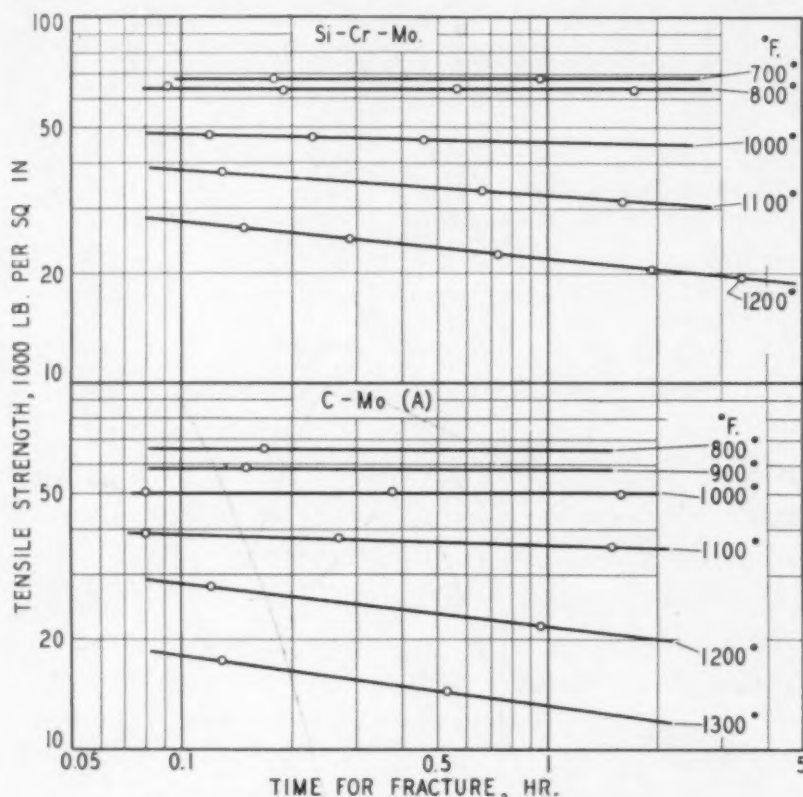


Fig. 5—Tensile Strength Versus Time for Fracture.

line relationship exists for each analysis at each of the temperatures considered. As before, the logarithmic lines are horizontal at the lower temperatures, thus indicating that the tensile strength value is not influenced, at least as the time for fracture is extended from approximately 0.05 to 2.0 hours. At the higher temperatures these lines assume a definite slope with the tensile strength values decreasing as the time for fracture is extended. The temperature at which these lines assume a definite slope differs with each analysis being a minimum with the open-hearth carbon steel and a maximum for the CMo (B) alloy.



### Determination of Critical Temperatures

While it is apparent from the curves of the first six figures that each of the analyses considered requires a different temperature for the slope of the logarithmic lines to change from zero to either a positive or negative value, they indicate a temperature range rather than a definite value.

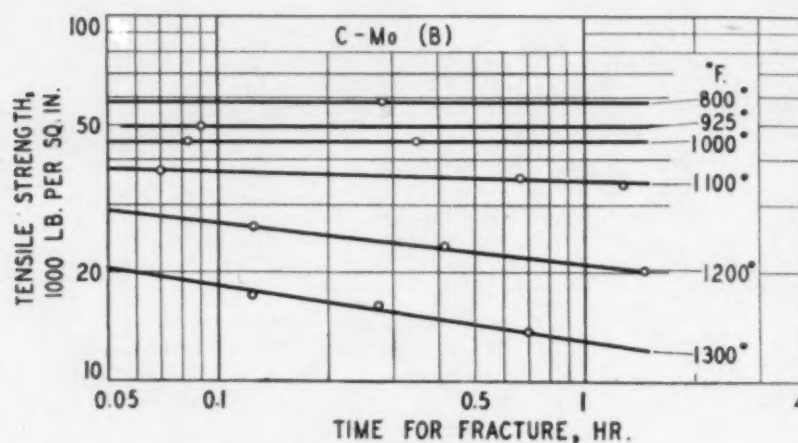


Fig. 6—Tensile Strength Versus Time for Fracture.

In order to show this change more exactly, results taken from these previous figures are replotted to semi-logarithmic co-ordinates in Figs. 7 to 11, inclusive. Two methods were employed. In the first, the tensile strength at each temperature corresponding to two different rates of deformation was plotted against temperature. The second method differed only in that two constant fracture times, rather than deformation rates, were considered. The same type curves result in both cases.

In all cases the results at the lower temperatures are represented by a single line. This is true since at these temperatures the tensile strength is not influenced by either the rate of deformation or the time required for fracture, at least over the range of deformation rates and fracture times considered. At the more elevated temperatures, however, the slope of the line varies depending on the deformation rate or the fracture time considered, and a separate line results for each condition.

As is evident from these five figures, the three lines, one representing the results at the lower temperature and the other two at the higher temperatures, intersect at a common point. This same condition is likewise true regardless of the number of deformation rates

or fracture times used. Two were employed in all of these figures merely for convenience and to maintain clarity.

There are two points of interest in these figures. The first is that the temperature, corresponding to the point of intersection, is

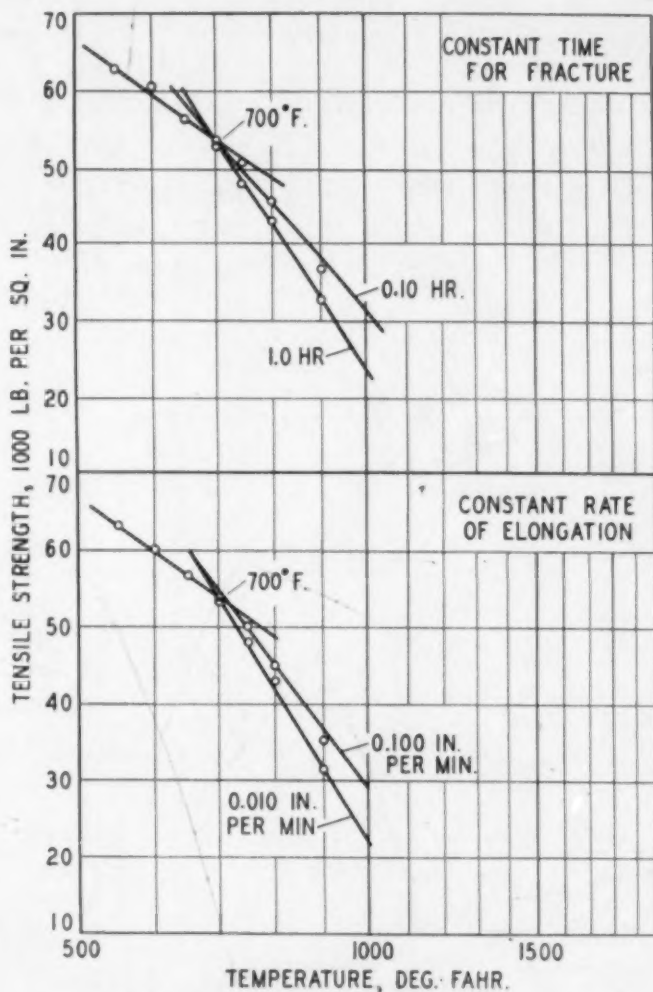


Fig. 7—Tensile Strength Versus Temperature for 1015 Electric Furnace.

the same regardless of whether deformation rates or fracture times are used. The only difference occurred in the CMo (A) steel and this amounted to but 5 degrees Fahr. From an experimental standpoint, therefore, the use of fracture times, rather than deformation rates, would be preferable since they are not only much easier to obtain but are also less open to argument.

The second fact of importance is that this temperature of intersection varies with each of the steels considered as is evident from the following values:

Steel	Temperature at Intersection Degrees Fahr.	
	Deformation Rate	Fracture Time
1015 (E1)	700	700
1015 (OH)	640	640
Si-Cr-Mo	890	890
C-Mo (A)	980	975
C-Mo (B)	1040	1040

The proposed method does, therefore, afford a means of classifying steels, but the question then arises as to whether these differences are in any way related to the creep characteristics.

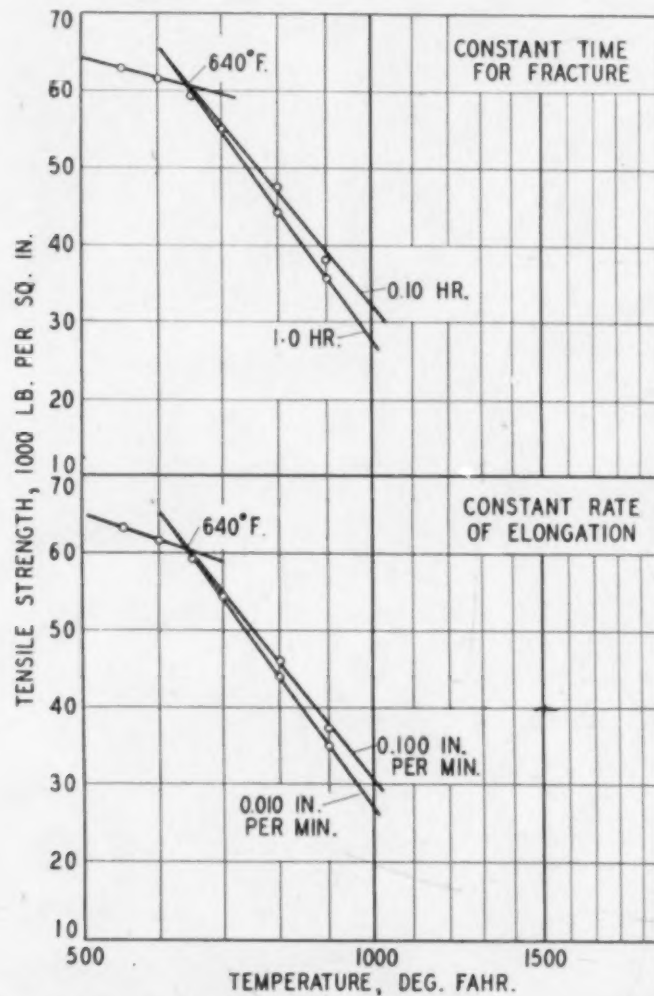


Fig. 8—Tensile Strength Versus Temperature for 1015 Open Hearth.

### *Critical Temperature Versus Creep Characteristics*

Creep results are available for four of the five analyses considered and these are summarized in Table I. The results are based



on tests of at least 1000 hours duration which were conducted in accordance with the A.S.T.M. Tentative Specimen E22-35T. Values are given for creep rates of 0.01 and 0.10 per cent per 1000 hours.

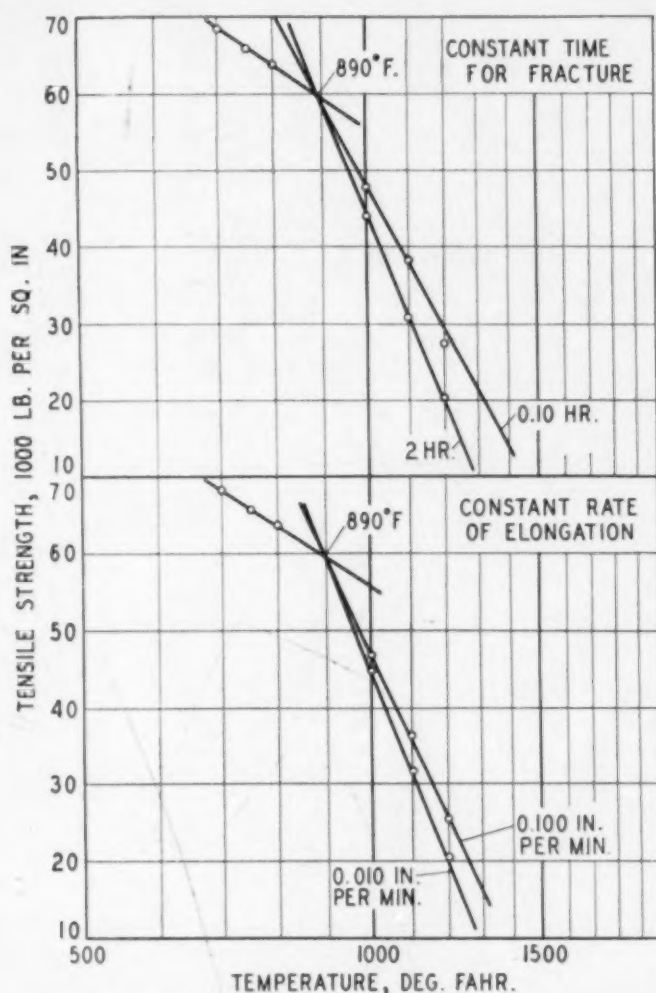


Fig. 9—Tensile Strength Versus Temperature for Si-Cr-Mo.

If the creep values at 1000 degrees Fahr. (540 degrees Cent.) be first considered it will be found that good agreement exists between them and the apparently critical temperature obtained from the tensile tests. The open-hearth S.A.E. 1015 steel possesses the lowest creep resistance and also the lowest critical temperature while the CMo (A) is superior in respect to both of these temperatures. The remaining two steels likewise fall into the proper order. In fact, as indicated in Fig. 12, a fairly straight-line relationship exists when creep strength and this critical temperature are plotted to logarithmic co-ordinates.

Table I  
Creep Characteristics at Indicated Temperatures for Four Designated Steels

Steel Designation	Temperature Degrees Fahr.	Stress for Designated Creep Rate Rate = per cent per 1000 Hours	
		0.01	0.10
1015 (OH)	800	12,000	17,200
	1000	1,800	3,300
	1200	140	540
1015 (El)	800	18,500	26,800
	900	12,800	16,900
	1000	2,700	5,750
	1200	290	620
Si-Cr-Mo	800	22,000	27,000
	1000	6,100	9,300
	1200	1,000	2,325
0.50 Mo (A)	800	15,500	26,000
	1000	10,700	17,800
	1200	495	2,000

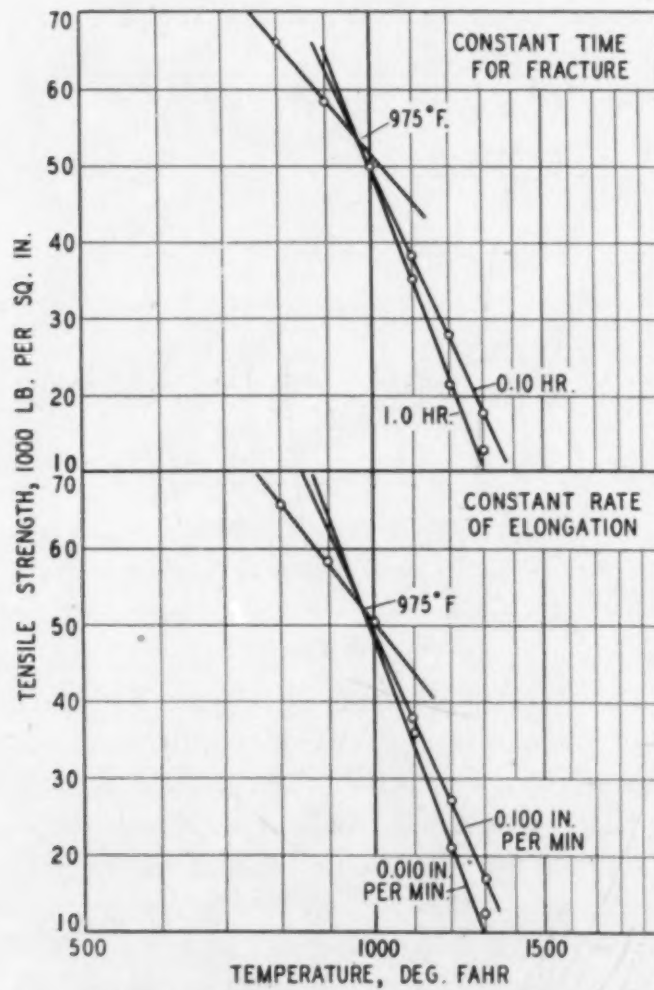


Fig. 10—Tensile Strength Versus Temperature for C-Mo (A).

At 800 degrees Fahr. (430 degrees Cent.) this same general relationship exists between the carbon steels but it does not apply to the remaining two analyses. In the case of the two alloy steels, however, 800 degrees Fahr. (430 degrees Cent.) is decidedly below the

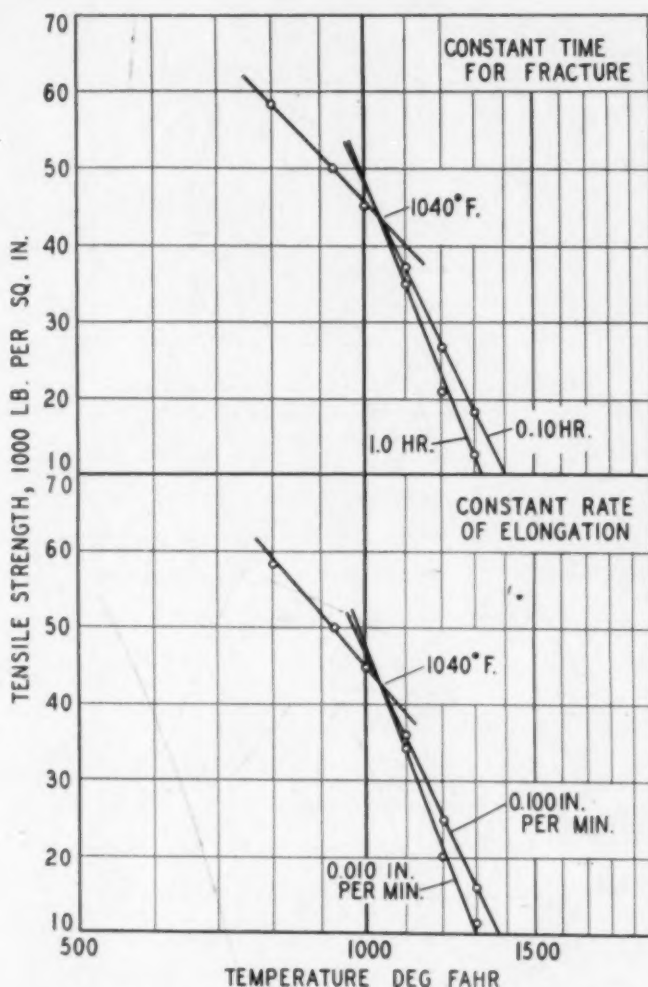


Fig. 11—Tensile Strength Versus Temperature for C-Mn (B).

critical temperature and thus the resulting creep characteristics depend to a very large extent upon the relative strain-hardening rate at the given temperature. The critical temperature on the other hand is believed to indicate, not relative degrees of strain-hardening at a given temperature, but rather the temperature above which strain-hardening is no longer of as great importance. The lack of agreement in the two alloy steels at 800 degrees Fahr. is not therefore surprising.

Likewise the results from the two types of test are in agreement



at 1200 degrees Fahr. (650 degrees Cent.) for the carbon steels but not for the two alloy steels. This temperature, however, is relatively far above the strain-hardening range and the rate of recrystallization

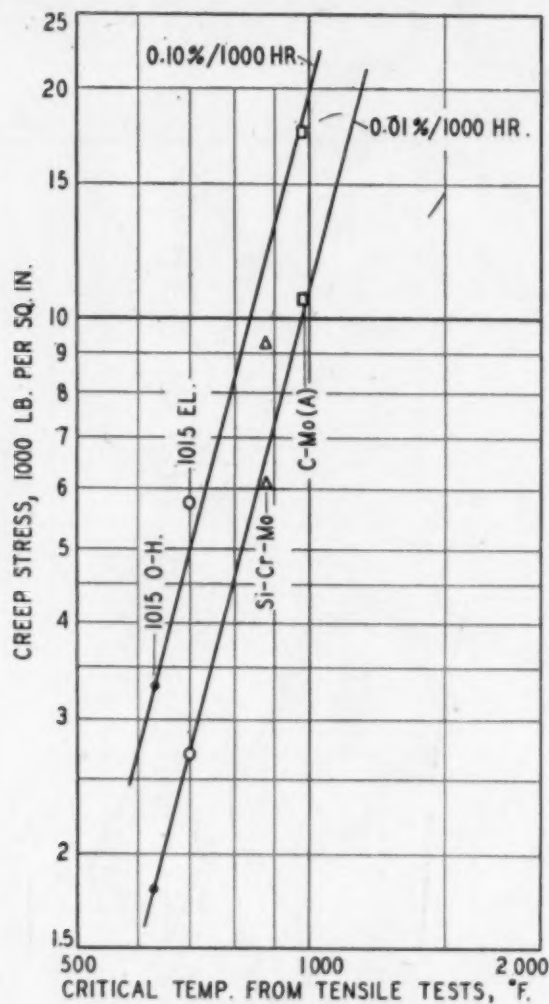


Fig. 12—Critical Temperature Versus Creep Strength at 1000 Degrees Fahr.

is dependent upon composition. Again this critical temperature is not believed to be an indication of differences in recrystallization rates but, as previously stated, an indication of the temperature below which recrystallization is not of as great importance.

On the basis of these results it appears that a relationship may exist between creep strength and the critical temperature obtained from the tensile tests but only at temperatures in the vicinity of this critical temperature. At temperatures below this, the creep characteristics are dependent on differences in the strain-hardening characteristics while at temperatures above, differences in the recrystallization

rates are the deciding factor. It is possible, however, that for steels of the same general type, such as the two carbon steels, this relationship may be found to apply over a much greater temperature range.

### CONCLUSIONS

On the basis of the results presented it is evident that the procedure outlined does differentiate between various analyses in that different critical temperatures were obtained for the steels considered.

Furthermore it appears that over a certain temperature range the creep characteristics are proportional to the magnitude of this critical temperature. In other words, the higher this temperature, the greater the creep resistance. The results further indicate that the temperature range over which this relationship applies may be further extended when similar analyses are being considered.

Even though the relationship between this temperature and the creep characteristic does not hold over the complete temperature range, it is believed that the proposed procedure may have merit as a type of acceptability test in indicating the relative creep resistance of different heats of the same analysis. Additional results will, of course, have to be accumulated before too definite claims can be made in this respect.

### ACKNOWLEDGMENTS

The authors wish to express their appreciation to H. H. Timken, Jr., of the Timken Roller Bearing Co., and to R. L. Wilson, formerly of the Timken Roller Bearing Co., and now with the Climax Molybdenum Co., for furnishing the necessary steels and for their permission to publish the creep values.

### DISCUSSION

**Written Discussion:** By P. E. McKinney, metallurgical engineer, Bethlehem Steel Co., Bethlehem, Pa.

The inconvenience of the long-time creep test as an acceptance standard for steels in high temperature service has long been admitted. Only recently, however, have we begun to recognize its inadequacy from the standpoint of yielding results applicable to all heats of a given grade. The authors have mentioned both points in their introduction, but I believe that they warrant further emphasis because in the light of these considerations, the paper becomes highly significant. One can scarcely over-rate the importance of developing a quick, dependable and reasonably accurate method for predicting the behavior of steels in high temperature service.

The authors do not presume, to be sure, that their procedure will com-

pletely fulfill this need. They conclude that it "may have merit as a type of acceptability test in indicating the relative creep resistance of different heats of the same analysis. Additional results will, of course, have to be accumulated before too definite claims can be made in this respect." Designing engineers have been too prone to consider the creep strength of a material as its sole requisite for use at high temperatures. Other characteristics such as stability of structure and resistance to embrittlement, also contribute in a large measure to its performance. This, too, has not been overlooked by the authors, but in the enthusiasm with which their paper will be received, there may be some danger in this direction.

It will be to the interests of the entire industry to undertake as promptly as possible and in a sufficient number of laboratories, an investigation of the possibilities and limitations of the method. Comparison of results with those of long-time creep tests will be essential. However, it is quite likely that some laboratories are in possession of materials from heats, the creep properties of which have already been determined. These should, in many cases, afford an excellent opportunity to prove the reliability of the procedure without the necessity of undertaking concurrently a time-consuming series of creep tests.

When it has been definitely established that the critical temperature of a steel, as determined by the short-time high temperature tensile test, constitutes an accurate index to its creep properties in the vicinity of that temperature, it will be desired, naturally, to put the procedure into immediate operation as an acceptance test. Before producers agree to this, however, I believe they should insist upon an opportunity to determine to what extent the critical temperature will vary from heat to heat in a given grade and to discover, if possible, the factors controlling such variation.

The authors have certainly merited the appreciation of manufacturers and users of high temperature steels for this effort to solve a problem which has troubled both groups for a long time.

#### **Authors' Reply**

We greatly appreciate the comments which Mr. McKinney has presented and we fully agree that too definite conclusions are not permitted at this time as to the suitability of this test as an acceptance test. There is no question, however, but what this testing procedure does differentiate between different heats in that a certain definite critical temperature does appear to exist for each analysis. If further work, both in our own laboratories and in others, shows that a relationship does exist between this critical temperature and the creep characteristics, then this test should have considerable value in indicating the high temperature characteristics to be expected from a given heat of steel.



## THE EFFECT OF GRAIN SIZE ON THE OXIDATION OF A LOW CARBON STEEL

By C. A. SIEBERT AND CLAIR UPTHEGROVE

### *Abstract*

*This paper deals with the effect of grain size on the oxidation of low carbon steels in the temperature range 1700 to 2100 degrees Fahr. It has been found that the fine-grained steels oxidize to a greater extent than coarse-grained steels. The ferrous iron content of the scale found at any given temperature in this range is higher for the fine-grained steels than for the coarse-grained steels, which results in a greater rate of diffusion and therefore a higher scaling loss.*

A PAPER presented by the authors (1)<sup>1</sup> at the sixteenth annual convention of the American Society for Metals gave data on the effect of temperature, time and partial pressure of oxygen on the scaling of S.A.E. 1020 steel. It was pointed out at that time that certain discrepancies were noted in the absolute numerical values of scaling between different S.A.E. 1020 steels. The present research was undertaken to find some explanation for these differences.

### LITERATURE AND METHOD

The literature on the subject of oxidation has been reviewed in previous publications from this laboratory by Murphy, Wood, and Jominy (2), Upthegrove and Murphy (3), and Siebert and Upthegrove (4); no review will therefore be given at this time. Likewise the experimental method has also been presented in these same papers. In the first part of this work, the samples were brought to temperature in an atmosphere of nitrogen, to prevent oxidation during the heating-up period. Dried and purified air was then passed through the furnace for one hour after the steel had reached the

<sup>1</sup>The numbers appearing in parentheses refer to the bibliography appended to this paper.

A paper presented before the Nineteenth Annual Convention of the Society held in Atlantic City, October 18 to 22, 1937. Of the authors, C. A. Siebert is assistant professor of metallurgical engineering and Clair Upthegrove is professor of metallurgical engineering at the University of Michigan, Ann Arbor, Michigan. Manuscript received June 3, 1937.

temperature of the furnace. In the second phase of this work the sample was introduced into the furnace, through which the stream of dried and purified air was passing, the total time in the furnace being one hour. The drying and purification of the air was accomplished

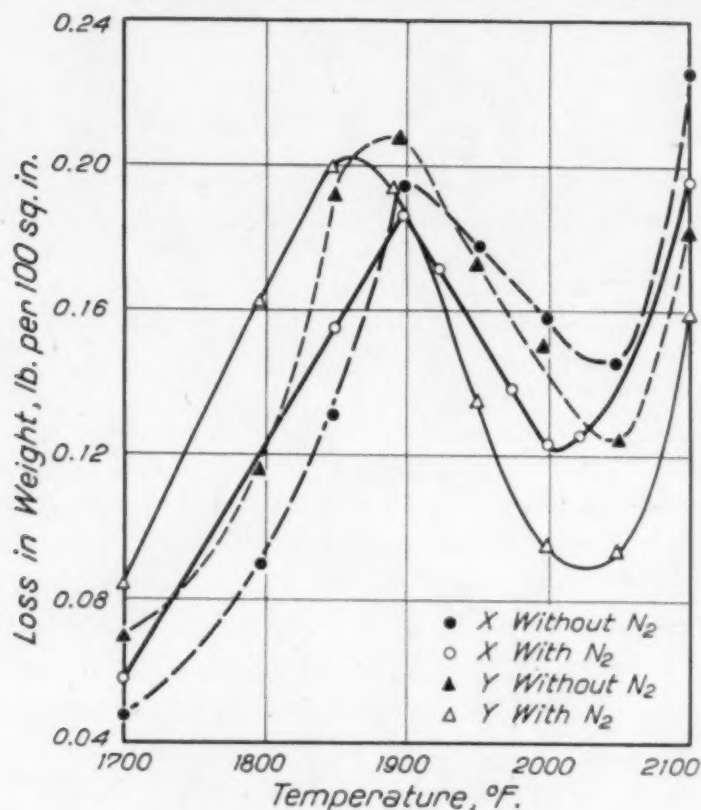


Fig. 1—Temperature vs. Loss in Weight.

by passing it through towers of calcium chloride, ascarite, and dehydrate. The analysis of the steels used was as follows:

	X	Y	
Carbon .....	0.15	0.18	Per Cent
Manganese .....	0.50	0.62	Per Cent
Silicon .....	0.23	0.23	Per Cent
Sulphur .....	0.032	0.023	Per Cent
Phosphorus .....	0.025	0.015	Per Cent
McQuaid-Ehn Grain Size ...	4-5	7	

### RESULTS

The scope of this investigation was limited to the effect of grain size on the oxidation of S.A.E. 1020 steel.

The temperature used varied from 1700 to 2100 degrees Fahr. The data obtained for both conditions, that is of scaling and not scaling when coming to temperature, are summarized in Fig. 1.

All of the curves show regions where the amount of scaling is

actually lowered with increasing temperature. This is in accordance with the work of Upthegrove and Murphy (5) and Siebert and Upthegrove (6). As pointed out in the previous publications, this decrease in scaling with increasing temperature is due to a difference in the degree of oxidation of the scale itself. As the temperature at which maximum scaling occurs is exceeded, an increasing proportion of ferric iron in the scale is observed. This increase in ferric iron content changes the character of the membrane through which diffusion of the oxygen, in some form, must take place. It also changes the solubility of oxygen in the scale, as FeO has a wider solubility range than  $\text{Fe}_3\text{O}_4$  which alters the driving force (concentration gradient) in the diffusional process. A definite relationship (7) is observed between ferrous iron content of the scale and loss in weight.

In the present work the steel having a McQuaid-Ehn grain size 4-5 has been designated as X and the one with a 7 grain size as Y. Comparing first the curves (Fig. 1) of the two steels raised to temperature in an atmosphere of nitrogen, it can be seen that the Y steel reached a maximum scaling loss at approximately 1865 degrees Fahr. and a minimum at 2025 degrees Fahr. The X steel reached a maximum at 1900 degrees Fahr. and a minimum at 2010 degrees Fahr. The Y steel scaled to a greater extent than the X steel at temperatures below 1925 degrees Fahr. Above 1925 degrees Fahr. the X steel oxidized to a greater extent than the Y steel.

In the case where the steel was allowed to scale while coming up to temperature, the Y steel reached a maximum at 1900 degrees Fahr. and a minimum at 2050 degrees Fahr. The X steel went through a maximum at 1900 degrees Fahr. and a minimum at 2050 degrees Fahr. The Y steel under this latter condition oxidizes more than the X steel below 1940 degrees Fahr., and less above this temperature.

Analyses for ferrous iron from oxides formed at 1800 degrees Fahr., when scaling was allowed during the heating-up period, gave values of 50.7 per cent ferrous iron for the Y steel and 44.1 per cent ferrous iron for the X steel. The curves Loss in Weight vs. Temperature for these conditions (Fig. 1) showed a value of 0.116 pound per 100 square inches for the Y steel and 0.09 pound per 100 square inches for the X steel. This again demonstrates that at any given temperature a greater FeO content of the scale will result in a greater loss in weight.



Comparing the X steel at 1800 degrees Fahr. under the two conditions, of scaling and not scaling while coming to temperature, it was found that the ferrous iron content of the oxide when nitrogen was used was 45.2 per cent while in the other case, where nitrogen was not used, it was 44.1 per cent. The losses in weight for the two conditions were 0.12 and 0.09 pounds per 100 square inches respectively. These values of scale analysis and loss in weight show two things: (1) the condition giving a higher ferrous iron content again resulted in a greater scaling loss, and (2) the scale formed during the heating-up period influences the types of membrane. When operating at temperatures below that at which the maximum ferrous iron content is formed, the effect of the prescaling while heating up will be to produce a lower FeO content in the scale than would have resulted if a nonoxidizing atmosphere had been used during the preheating treatment. Therefore, the scaling loss will be less for the case where the steel is heated up in an oxidizing atmosphere when operating at temperatures below that at which the scale forms a maximum FeO content, than where a nonoxidizing atmosphere when operating at temperatures below that at which the scaling at temperatures above that at which the maximum FeO content is obtained, the scale formed while the steel is coming to temperature will have a higher FeO content than it would have had if oxidation had begun at any given temperature in this range. Therefore, the scaling loss will be less when operating at temperatures above that at which the maximum FeO content occurs, for the case where the steel is heated up in a nonoxidizing atmosphere than where oxidation is allowed during the heating-up period. This can be seen from the curves of Fig. 1 where the steels heated to temperature in a nonoxidizing atmosphere scale less above 1850-1900 degrees Fahr. than the same steels allowed to oxidize during the preheating time. The maximum for the curve Ferrous Iron Content vs. Temperature always occurs at a temperature slightly below that at which the maximum occurs in the Loss in Weight vs. Temperature curve. This is due to the fact that the coefficient of diffusion increases with increasing temperature, while at the same time the driving force is becoming somewhat less due to the formation of a greater amount of  $\text{Fe}_3\text{O}_4$  which has a lower solubility of oxygen and therefore a small concentration gradient. It is evident that the two effects are opposing each other and for a limited temperature increase the effect of increasing the coefficient must be the stronger of the two.

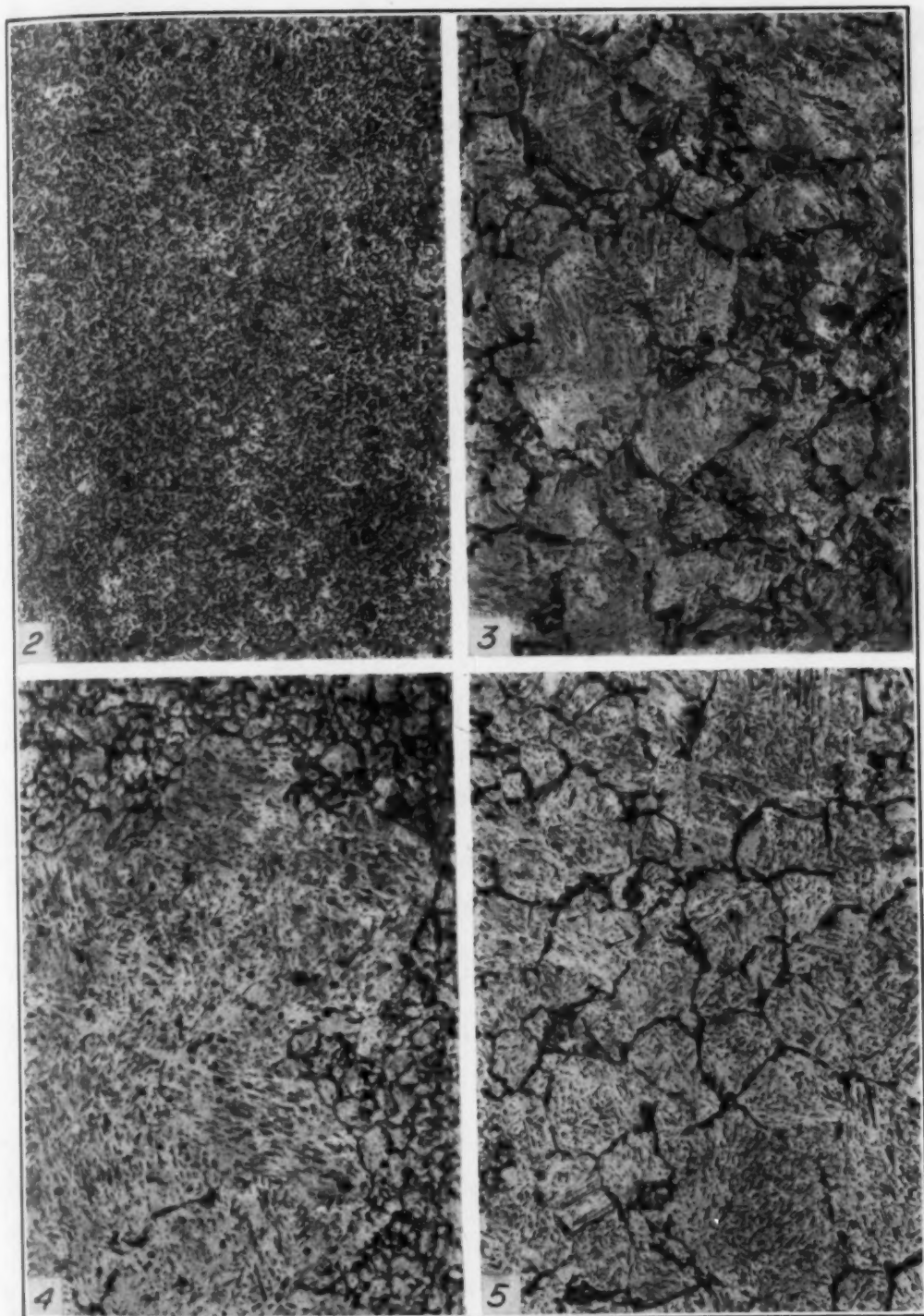


Fig. 2—Y Steel. 1700 Degrees Fahr.  $\times$  100.  
Fig. 3—X Steel. 1700 Degrees Fahr.  $\times$  100.  
Fig. 4—Y Steel. 1800 Degrees Fahr.  $\times$  100.  
Fig. 5—X Steel. 1800 Degrees Fahr.  $\times$  100.

With further increase in temperature the effect of the higher degree of oxidation of the scale predominates over the change in the diffusion coefficient.

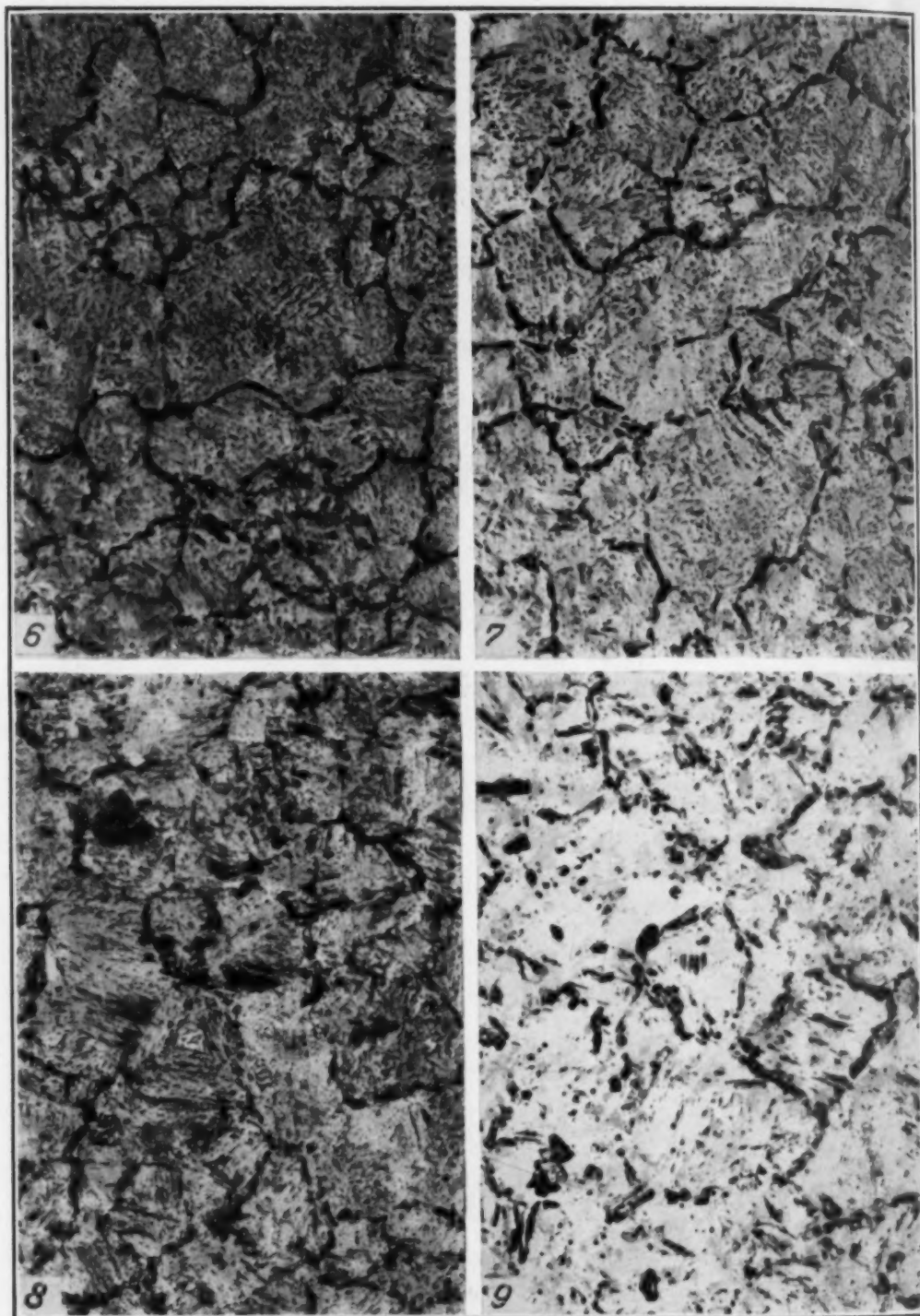


Fig. 6—Y Steel. 1900 Degrees Fahr.  $\times 100$ .  
Fig. 7—X Steel. 1900 Degrees Fahr.  $\times 100$ .  
Fig. 8—Y Steel. 2100 Degrees Fahr.  $\times 100$ .  
Fig. 9—X Steel. 2100 Degrees Fahr.  $\times 100$ .

One factor which may affect the absolute numerical values of scaling at any given temperature is the grain size of the steel at that temperature. The grain size of the steels used in this investi-



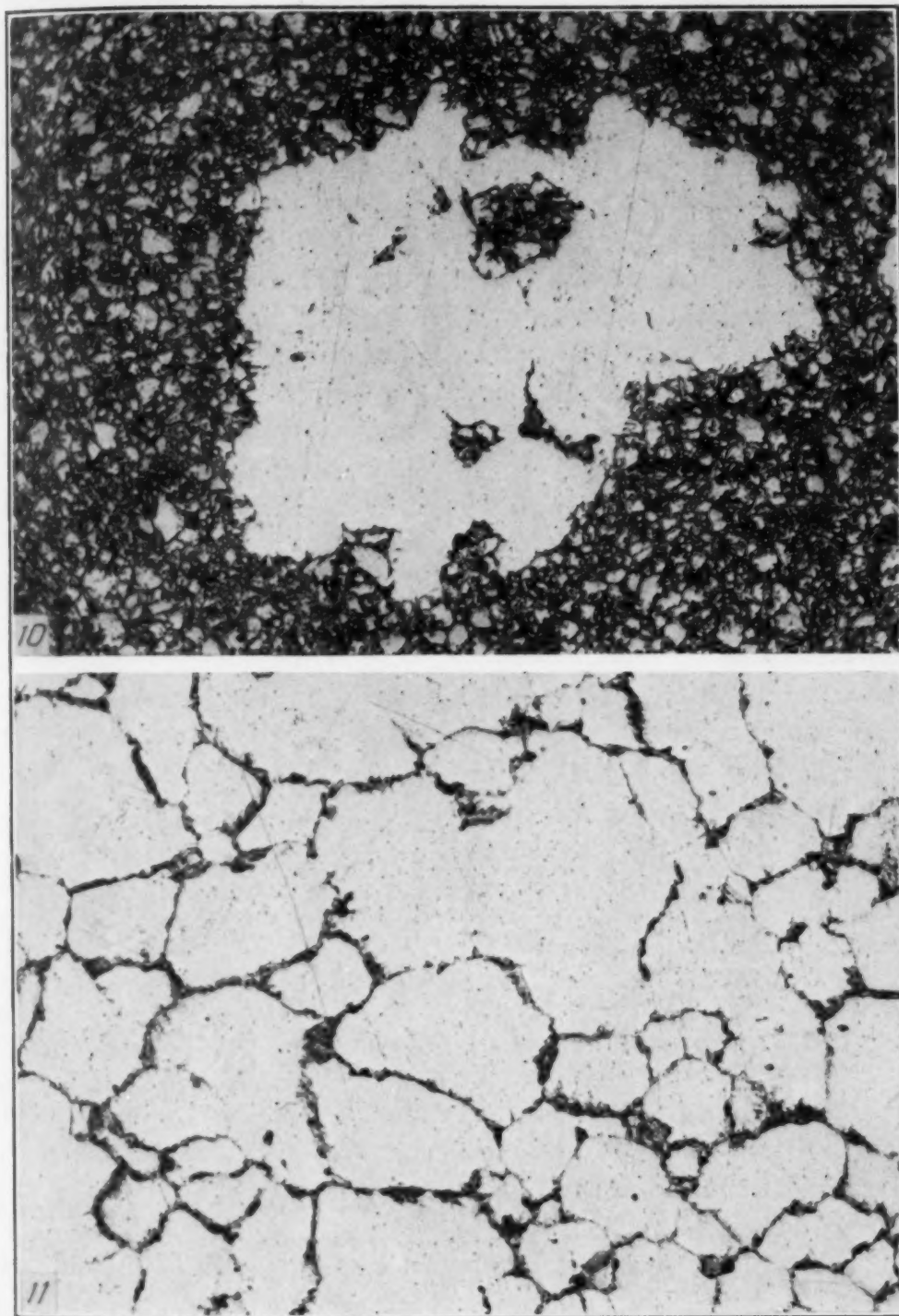


Fig. 10—Y Steel. Coarsened to This Grain Size and Scaled at 1700 Degrees Fahr.  
Fig. 11—Y Steel. Coarsened to This Grain Size and Scaled at 1700 Degrees Fahr.  
Magnification of both Photomicrographs  $\times 100$ .

gation was run at temperatures from 1700 to 2100 degrees Fahr. This was accomplished by heating the steels for one hour at the temperature at which it was desired to establish the grain size, cool-

ing to 1700 degrees Fahr., and quenching in water. This method was used instead of carburizing because the latter method does not always establish the correct coarsening temperature.

Figs. 2 to 11 show the results obtained at the various temperatures. At 1700 degrees Fahr. the Y steel is fine-grained while the X steel is coarse. At 1800 degrees Fahr. the Y steel has a very mixed grain, some areas being very fine while others are very coarse (Fig. 4). The X steel at 1800 degrees Fahr. shows practically no growth over the grain size at 1700 degrees Fahr. At these temperatures, 1700 and 1800 degrees Fahr., the X steel scales less than the Y steel. At 1900, 2000, and 2100 degrees Fahr. the Y steel is coarser grained than the X steel, and at these temperatures the scaling loss is also less for the Y steel than for the X steel.

Since variations in scaling may be attributed to other factors than differences in grain size, it appeared desirable to make additional scaling tests in which but one steel with varying grain size was used. The Y steel was chosen for these experiments because of its fine grain size at 1700 degrees Fahr. and the coarsening effect at higher temperatures. The method involved heating the steel at temperatures above 1700 degrees Fahr. for thirty minutes in an atmosphere of dried and purified nitrogen, lowering the temperatures to 1700 degrees Fahr. in this same atmosphere, and then pass-

Table I

Grain Size	Loss in Weight Pounds Per 100 Square Inches
Fig. 2 .....	0.081
Fig. 10 .....	0.072
Fig. 11 .....	0.064

ing the dried and purified air through the furnace for a one-hour period. The results are given in Table I. These results show that increasing the grain size resulted in a lower scaling loss at 1700 degrees Fahr.

The authors feel that other factors than grain size influence the absolute numerical values of scaling. The decarburization which occurs along with oxidation may have a decided bearing on scaling. This will be considered in another paper on "The Effect of Carbon Content on Oxidation of Steel," to be presented in the near future.

Acknowledgment is made to R. L. Leslie for determining the chemical analyses of the scale.

## References

1. C. A. Siebert and Clair Upthegrove, "Oxidation of a Low Carbon Steel in the Temperature Range of 1650 to 2100 Degrees Fahr.," *TRANSACTIONS, American Society for Metals*, 1935, Vol. 23, p. 187.
2. D. W. Murphy, W. P. Wood, and W. E. Jominy, "Scaling of Steel at Elevated Temperatures by Reaction with Gases and the Properties of the Resulting Oxides," *TRANSACTIONS, American Society for Metals*, 1932, Vol. 19, p. 193.
3. Clair Upthegrove and D. W. Murphy, "Scaling of Steel at Heat Treating Temperatures," *TRANSACTIONS, American Society for Metals*, 1933, Vol. 21, p. 73.
4. C. A. Siebert and Clair Upthegrove (*Ibid*).
5. Clair Upthegrove and D. W. Murphy (*Ibid*).
6. C. A. Siebert and Clair Upthegrove (*Ibid*).
7. C. A. Siebert and Clair Upthegrove (*Ibid*).

## DISCUSSION

**Written Discussion:** By W. E. Jominy, Research Laboratories Division, General Motors Corp., Detroit.

The experimental procedure in this paper leaves very little room for doubt of the conclusions reached and the authors deserve congratulations for their work. It is unexpected to find the difference in scaling due to grain size as great as shown in Fig. 1. Would the authors think this might be a contributing factor in the relative hardenability of the coarse-grained steel compared to the fine? I would not expect this to be a major factor but it might have some effect which the authors who have the steel and equipment available might easily measure.

The statement that decarburization may have some effect on scaling certainly appears well justified. It is also likely that the reverse of this is true, that is, the type of scale formed affects the decarburization. These two phenomena are so closely associated that it has always been difficult to study the one without considering the other. It would be very worth while to know whether certain compositions of scale inhibit decarburization. If the authors can throw some light on this complex problem we will be further indebted to them.

**Written Discussion:** By John A. Webber, production manager, Interstate Drop Forge Co., Milwaukee.

It is gratifying to know that the authors have continued with their investigation of oxidation and have included the effect of grain size. This paper indicates that a steel with a large grain size at the temperature of oxidation does not lose as much metal as does a steel which is fine-grained. We would all, of course, like to know why this is the case. The authors may have a theory for explaining this and I am sure we would all be interested in hearing it. One possible explanation might be that in a coarse-grained steel a smaller percentage of the surface is made up of grain boundaries. At forging temperatures we know that the scale is formed deeper at the grain boundaries than in the grain itself. It would therefore seem logical for fine-grained steel to lose more weight in scaling than coarse-grained steel because a larger part of the surface is made up of grain boundaries.

The writer will look forward to reading the authors' future paper on



"The Effect of Carbon Content on Oxidation of Steel." It seems evident that the type of scale formed has a bearing on the amount of decarburization under the scale and it is hoped that their investigation will shed some new light on this complicated problem.

**Written Discussion:** By R. H. Harrington, research metallurgist, General Electric Co., Schenectady, N. Y.

The authors have contributed another interesting paper to the list of valuable publications by the metallurgists of the University of Michigan concerning the scaling of steel.

While it is agreeable to refer to previous publications for the experimental method, it would add interest to the present paper to include the shape and dimensions of the test samples. The writer believes that the curves "without  $N_2$ " and "with  $N_2$ " (Fig. 1) should not be directly compared in a quantitative manner since apparently the oxidization-exposure conditions are not identical as to time at temperature. The qualitative comparison of these curves is interesting as there are the marked differences noted by the authors.

From the viewpoint of the title of the paper, the writer found most interest in the effect of different grain sizes in the same steel as shown in Table I. It should be of interest to repeat similar experiments on steels of various carbon contents and a few alloy steels in order to establish the validity of a general conclusion. It is of interest to note that the authors are studying the effects of inherent factors other than grain size.

#### Oral Discussion

R. W. SANDELIN:<sup>1</sup> This problem of the scaling of steel is of particular interest to us, because we make a number of products where the retarding of scaling is of the utmost importance; I have in mind particularly the manufacture of steel hoops for cooperage purposes. It seems that it is a very desirable thing to the trade to keep down the amount of scale on this type of product because in putting the hoops over the barrels, a hoop that has a good deal of scale on it tends to smudge the barrel. That may not seem to be very important to some users of hoops, because these barrels, after they are filled with liquor, are put in a warehouse and then aged; nevertheless a good many consumers of this product want a steel hoop that is fairly free from scale. We have studied this problem for some time and find that there are a good many things other than grain size that affect the amount of scale on the final hoop. We found in our studies that the grain size varies considerably, and it seems that the heat of the steel rather than the grain size has a bearing on the amount of scale that will form; in other words, you may have quite a variation in grain size, ranging from seven to two, and find there is no consistent correlation between the grain size and the scale, but rather the amount of scale that forms seems to hinge largely on the heat of the steel, and the particular method of manufacture. I notice that the authors of the paper mention something about phosphorus having some effect. These hoop steels are rephosphorized and therefore are very high in phosphorus, sometimes as high as 0.085 per cent. We find that in a range of 0.025 to as high as 0.075 per

<sup>1</sup>Atlantic Steel Co., Atlanta, Ga.

cent, the phosphorus content seems to have very little effect with this type of steel. We have studied the effect of phosphorus and have come to the conclusion that it has very little to do with the amount of scale formed. We are still wondering what are the real factors affecting the amount of scale formed on steel hoops. The steel, incidentally, is delivered from the final finishing rolls at about sixteen hundred degrees Fahr. after being thoroughly scraped, and we find that we can notice, just by visual observation of the scrapers, whether one steel scales more than another. It seems to be an inherent quality not associated so much with grain size, but in other factors that we have not been able to determine entirely. In regard to the carbon content of these hoop steels, it is fairly high, ranging from 0.30 to 0.40 per cent carbon. For forming operations, punching, etc., the amount of scale should be kept to a minimum to keep down the wear on the dies. We are still looking for a complete solution to the problem, and while we have it worked out fairly well, we think there is a good deal more to be learned on this subject. I think the authors are to be congratulated however and hope they will continue their work.

W. P. DAVEY:<sup>2</sup> I should like to say something about the effect of grain size on the amount of oxidation you can get in a sheet of steel. We must assume that these grains are crystals of metal. Now these crystals of metal will have different orientations. There is no reason to assume that adjacent crystals will have identically the same orientations. The result will be something like what I shall put on the blackboard. I shall draw a fanciful picture of two crystals, one with an orientation like this, the o's representing individual atoms. Let us say we have another crystal with a different orientation and again let us use o's to represent the individual atoms. Now let those two crystals start to grow together. As the material solidifies there will be a region in which the atoms will tend to take intermediate positions between those locations required by one crystal and those required by the other crystal.

It is reasonable to assume that the energies of those atoms will be different from those of the normal atom inside the crystals themselves, so that it is to be expected that corrosion will take place primarily between the grains of the metal rather than in the body of the grains. Such a conclusion is consistent with ordinary metallurgical experience. We should therefore expect the oxidation of these sheets of steel to take place primarily between the grain boundaries. Obviously the smaller the grain size, the bigger the area of grain boundaries, and therefore it is reasonable to assume that the amount of oxidation will be a function of the particle size of the individual crystals. I think we should, then, assume that the dependence of oxidation on grain size is only a secondary dependence, that both the grain size and the degree of oxidation will depend upon the amount of intercrystalline boundary material.

C. A. SIEBERT: The discussion has been very gratifying, because I had hoped I would get some new ideas, which I did. Taking these comments in no particular order, first, I will attempt to answer the last question concerning the experimental procedure. The samples were cylinders two and a half inches long and five-eighths of an inch in diameter that had been polished with

<sup>2</sup>Research Professor, Pennsylvania State College, State College, Pa.

two zero emery papers to get a uniform surface. Most of the scale formed split off in the quenching bath. The small remnant of scale still adhering was disposed of in an electrolytic stripping bath using a sulphuric acid solution. Blanks were run to show to what extent the base metal would be affected, which was negligible. The scale which split off in the quenching bath was dissolved in sulphuric acid, titrated with permanganate, using a double titration to get both ferrous and ferric iron.

I would like to ask Mr. Sandelin whether or not his tests were run under controlled conditions, or whether the steel was placed in a furnace which was open to the atmosphere.

MR. SANDELIN: Well, most of our experiments were made under actual production conditions, right in the mill. It is not really a laboratory proposition with us since we have to produce this steel and sell it to the customers, therefore, our experiments were mill experiments. However we were continually checking back endeavoring to find out why some heats gave much better results than others. In that sense, we did a considerable amount of laboratory work.

MR. SIEBERT: In all our previous work, oxidation was accomplished by passing air that had been dried and purified of  $\text{CO}_2$  through the furnace. We have run some subsequent experiments and hope to have them ready for publication soon, regarding the effect of humidity, and we find that at certain temperatures the difference between dry air and air on a rainy day may make almost a hundred per cent difference in the amount of scaling. For that reason, unless tests are run under conditions where you are using the same atmosphere all of the time, you cannot very well draw any general conclusions. In regard to the discussion by Mr. Webber, we would like to say that we feel also that decarburization plays an important part along with oxidation. In that regard we are now making a study of higher carbon steels as was mentioned in the paper, and we have more or less formed a theory regarding their behavior. However, since all the results are not finished, I do not think it would be wise to go into that theory at the present time.

#### Authors' Closure

The authors are indebted to those who have contributed to the discussion of this paper.

They agree with Mr. Jominy that the type of scale formed affects decarburization, because the rate of diffusion of oxygen inward is affected by the type of scale which has been formed. Oxidation and decarburization always occur simultaneously, and it is impossible at this time to say whether or not a type of scale could be formed which would allow an oxidation rate which was equal to, or greater than, the rate of decarburization. Regarding Mr. Jominy's question on hardenability, we do not feel that the scaling characteristics of coarse- and fine-grained steels would be a major factor in the relative hardenability of the steels.

Mr. Webber calls attention to the fact that, at forging temperatures, oxidation proceeds more rapidly at the grain boundaries than in the grains themselves, and that the finer-grained metal has more grain boundary area than the coarse-grained steel. We have noticed this phenomenon at temperatures



below the forging range also, but we do not believe that the difference in grain boundary material in coarse- and fine-grained steels can account for the difference in scaling observed at any given temperature. However, the amount of grain boundary material is undoubtedly a contributing factor.

Mr. Harrington points out that curves "without  $N_2$ " and "with  $N_2$ " (Fig. 1) should not be directly compared in a quantitative manner because the oxidation-exposure conditions are not identical as to time at temperature. No quantitative comparison was intended because the time at temperature varied for the two conditions. However, the difference in absolute scaling values, for any given temperature for the two conditions of scaling and not scaling when coming to temperature, cannot be explained by the difference in time at temperature. The comparison made on page 1054 was only intended to be qualitative, to show the effect of the scale, formed while coming to temperature, on the oxidation of the steel when at temperature.

Mr. Sandelin states that his experience with the effect of phosphorus on the scaling of steel seems to indicate that this element does not affect the oxidation to any marked extent. The authors have not investigated this point, and their statements that phosphorus may affect the scaling of steel was based on the work of Scheid and Kiwit:\*

Professor Davey points out that the energy content of the atoms in the grain boundaries is different from the energy content of the atoms in the grains themselves, and that corrosion is favored to take place at the grain boundaries. While it may be assumed that the grain boundaries conditions offer a more favorable condition for the beginning of oxidation, it cannot be agreed that once a film of oxide is formed on the surface of the steel that oxidation continues primarily between the grains.

\*"Influence of Alloys on Scaling of Iron," *Archiv für das Eisenhüttenwesen* 9(8):405-416 (1936).

## FRICTIONAL WEAR TESTS OF LUBRICATED SURFACES

BY C. J. COBERLY AND FRED M. ARNOLD

### *Abstract*

*This paper describes a method for the rapid determination of the frictional wear of lubricated metal surfaces. The testing machine reciprocates test samples under load in an oil bath. The samples are prepared by lapping optically flat. The small amount of wear which occurs in a few hours is measured by the refraction pattern produced between the worn surface and an optical test plane. An electrical gage and the profilograph as supplementary methods of measuring wear are described. The results of wear tests are given. The method has been used to determine the comparative lubricating value of a few light refined oils. A table of materials tested and their Rockwell hardnesses is included.*

EXPERIENCE gained from the operation of an almost infinite number of machines enables designers to predict with certainty the wear resistance of certain alloys when run against each other. This experience has established what is good engineering practice. Using this store of information new machines are designed and built with confidence.

Vast and varied as are the number of machines in which frictional wear occurs, the number of couples with predictable behavior is small. Requirements for new mechanisms or improvements in old ones continually arise in which it is necessary to go beyond the borders of predictable behavior of the metals or to use newer alloys for which insufficient experience has yet been accumulated to predict their performance.

In the course of developing and manufacturing a hydraulic pump for oil wells, it became necessary to search for combinations of alloys with outstanding wear resisting properties. The pump is subjected to fluid operating pressures up to 10,000 pounds per square inch. Its dimensions are small, being limited by the tubing size in

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A paper presented at the Western Metal Congress held in Los Angeles, California, March 21-25, 1938. The authors, C. J. Coberly is president, and Fred M. Arnold metallurgist of Kobe, Inc., Huntington Park, California.

the well. Stresses in the parts are therefore high. In many cases the crude oils to be pumped have poor lubricating qualities and in addition may contain abrasives such as sand. The water usually associated with the crude oils are often corrosive. Some crude oils are themselves corrosive. The presence of water in the oil also accelerates the type of wear known as "wire drawing" or high pressure cutting; this cuts the pumps very badly.

Pumps constructed of conventional alloys failed to give satisfactory service. It was apparent that a systematic search for alloys with better wear resisting properties than those in use was necessary. This required standardizing on some method of wear testing to determine the suitability of a number of available alloys. Because of the many alloys and combinations of alloys which it would be desirable to test, the method chosen had to be rapid. It must also closely simulate the operating conditions of the pump in a well: That is to say, the wear of polished lubricated surfaces reciprocating under high pressures. The test results of the method adopted must be reproducible.

Since no existing wear tester met these requirements a special machine was designed for the purpose. This machine has now been in use for four years and more than three hundred tests have been run with it.

Most of the schemes proposed for testing resistance to wear are special methods, frequently only applicable to the special case under investigation. Metallurgists and engineers are generally familiar with these. We shall not classify and describe the different types of wear or different machines. This has been done in an excellent manner in the American Society for Testing Materials, Symposium on Wear of Metals,<sup>1</sup> recently published. This symposium also contains a comprehensive bibliography of one hundred thirty-seven references. We shall only quote here Gillett's definition of wear of a metal part as "its undesired gradual change in dimensions in service under frictional pressure."

#### TEST METHOD AND MEASUREMENT OF WEAR

*Method*—This method of wear testing consists of reciprocating sample blocks of the metals under investigation against each other under load. The tests are usually run in oil. The test blocks are

<sup>1</sup>Symposium on Wear of Metals, A.S.T.M., April 5, 1937.



lapped until they are optically flat except near the edges. (A few soft metals cannot be lapped.) They are put in the machine and run under load for a given time. They are then examined under an optical test plane. The amount and location of the wear which has occurred is shown by the refraction pattern. In this way the very small amount of wear which is produced in a few hours is measurable.

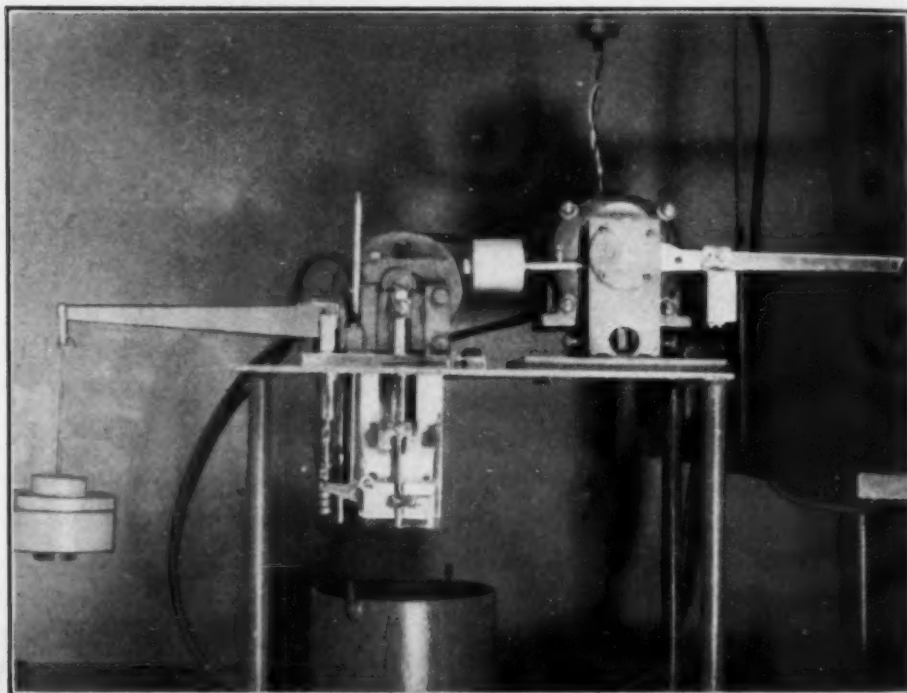


Fig. 1—Front View of Testing Machine Showing General Arrangement.

*Test Machine*—Fig. 1 shows the front side of the testing machine. The essentials of the machine are a test block holder, a motor-driven crosshead to reciprocate the movable center test block and the lever system for applying the load. The machine being used at present in this work is the third designed and built for the purpose. The improvements in design consist in changing the position of the samples from horizontal to vertical, improving the bearings and placing the bearings on top out of the crude oil bath.

Samples of the alloys to be tested are made into rectangular blocks finished 1.0 by 0.5 by 0.25 inches. Three specimens are tested simultaneously. Fig. 2 shows the sample holders. Two test blocks on edge are held in stationary holders with the long axis horizontal. These holders are  $\frac{3}{4}$  inch deep and are ground exactly

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0.5 inch high by 0.225 inch wide. With slight lapping of the edges for clearance the blocks are free to float parallel to the axis of loading in the holders. A clip across their ends holds the blocks in place. The third block reciprocates. It is held with the long

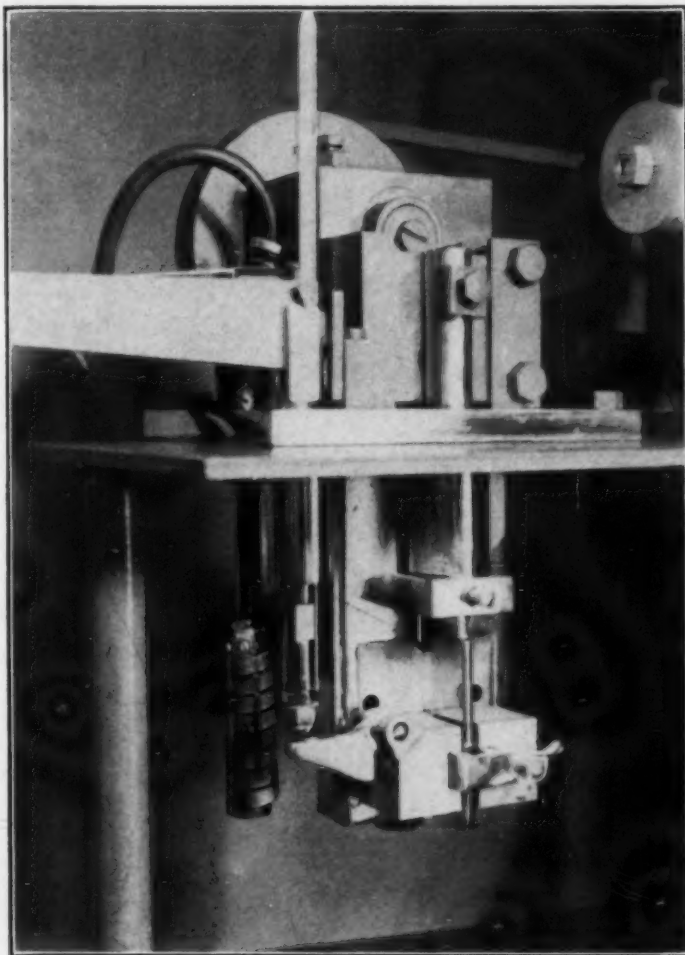


Fig. 2—View of the Sample Holder Showing Running Position.

axis of the block vertical in a holder with a 1.0 inch by 0.5 inch opening. Bolting this holder to the crosshead positions this center reciprocating block between the other two horizontal stationary blocks. This arrangement provides for testing four surfaces (two couples) simultaneously. The three test blocks can all be different if desired.

The holders are designed so that the center block contacts exactly the inner 0.5 inch of the outside pair so that the area of contact is 0.5 by 0.5 or 0.25 inch square inch. The crosshead which reciprocates the center block has a  $\frac{5}{16}$  inch stroke so there is a  $\frac{3}{16}$  inch

overlap which is not uncovered at the end of the stroke. The test load is applied to the outer or left hand block by a push rod with a rounded end (radius of end of push rod is  $\frac{3}{8}$  inch) which approximates point contact. As stated, the blocks are free to adjust themselves so that the faces are parallel, but for best results other motion should be avoided by accurately fitting the blocks to the holders. It is important that the faces of the blocks be parallel.

Fig. 3 is a schematic diagram of the testing machine. The load is applied through levers. Weights are hung at the end of an 8-inch lever of the second order. The load is transmitted through a long adjustable push rod to a second multiplying arm of the first order at right angles to the short push rod resting against the sample. Hardened and polished steel knife edges and bearing plates are used. The long push rod is adjustable to accommodate varying thicknesses of samples. A dynamometer is provided to measure the torque required to reciprocate the blocks under various conditions. Pressures up to 4000 pounds per square inch can be applied and have been used. A  $\frac{1}{4}$ -horsepower, 3-phase induction motor drives the countershaft by a V-belt drive. The pulleys provide a 3:1 reduction. The motor speed is 1475 revolutions per minute giving the countershaft a speed of 492 revolutions per minute or 984 reversals per minute. This results in a surface speed of 25.6 feet per minute for the test blocks. (This was chosen as being of the same order of speed as the piston travel in the hydraulic pump as originally designed.)

The speed varies somewhat with the loading, being approximately 984 reversals per minute at no load dropping to 950 reversals when the samples are loaded to 4000 pounds per square inch.

Ball bearings are used on all the shafts. A power take-off drives a stirrer to keep the test oils circulated. An emulsifying pump is provided for use in testing the effect of water in the crude oils. The test oil, about three pints, is contained in a small steel tank which is bolted on the bedplate and submerges the test blocks. An electric immersion heater and thermostat are provided for heating the bath and holding it at any desired temperature. The tank is not gas tight. See Fig. 1. Fig. 4 shows the back of the testing machine. The conditions under which the tests are run will be dealt with under the Results of Wear Tests.

*Preparation of the Test Blocks*—Samples from the metals to be tested are ground into rectangular blocks 1.0 by 0.5 by 0.25 inch.



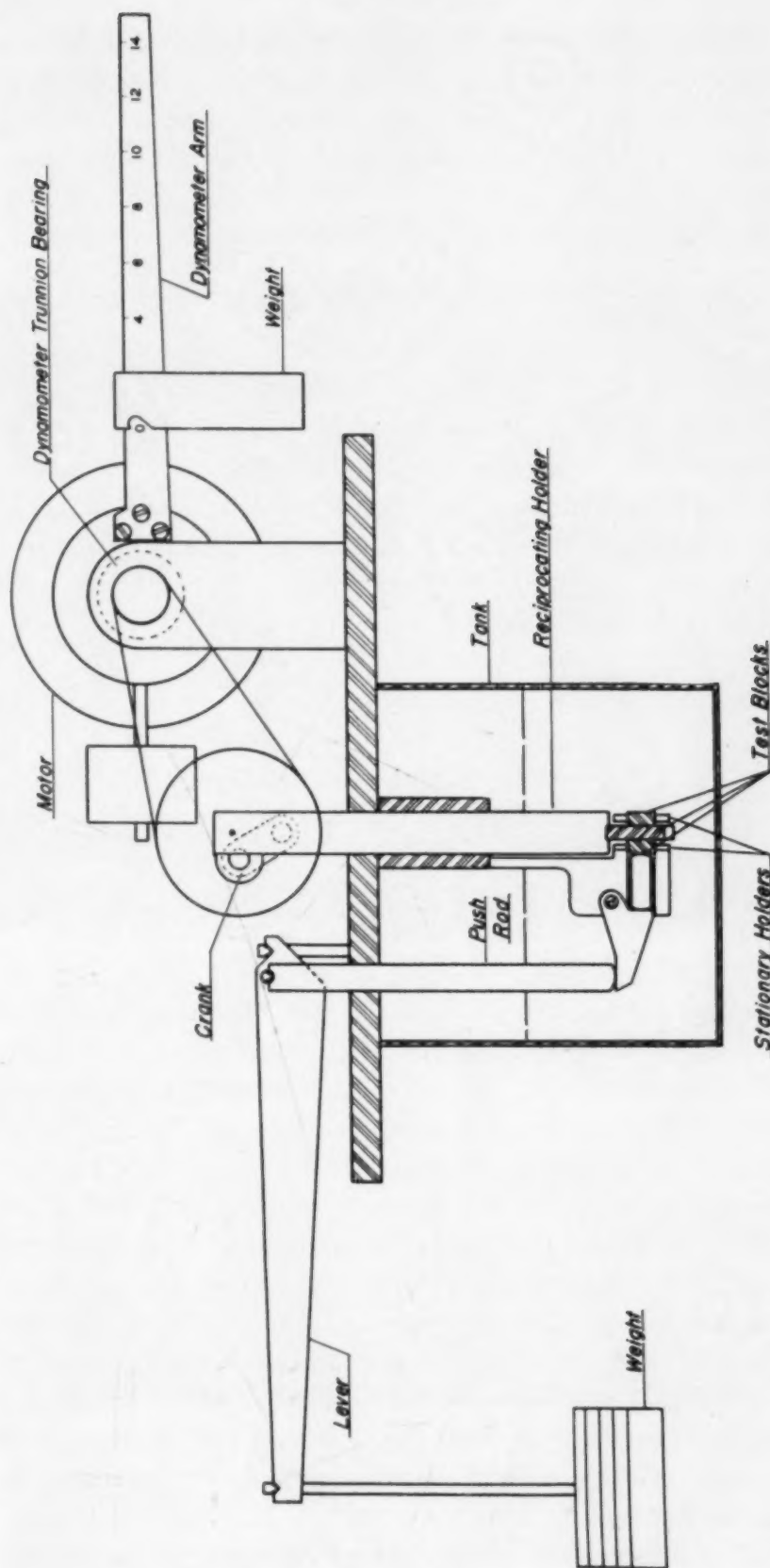


Fig. 3—Schematic Arrangement of the Elements of the Testing Machine.

When preliminary heat treatments are used proper allowance for finishing by grinding is made. The surfaces are ground exactly at right angles to each other. The length and width are held to within 0.0005 inch. The finishing wheel used is a 36T22 Aloxite wheel. The blocks are then hand lapped on a cast iron lap with 800-grain crystalon. They are finished by lapping by hand on a cast iron lap

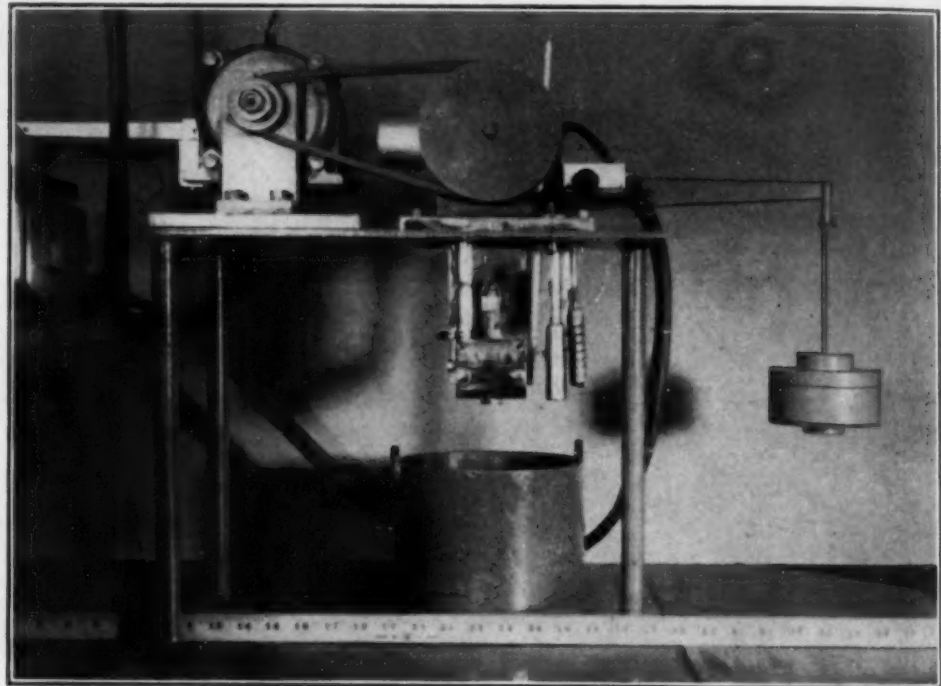


Fig. 4—Back View of the Testing Machine.

with 900-grain crystalon until they are optically flat. This simulates the lapped finishes of the pump parts. About twenty minutes are required to hand lap one surface. No difficulty is experienced in making the blocks flat to within two fringes at the ends. This is fortunately about as flat as they should be, as a slight rounding of about one-half a fringe at the edges promotes the entry of oil. It is possible to lap the blocks so flat that they gall when run together.

An important requirement in testing for wear is to be able to duplicate the finish on the wearing surfaces. This requirement has frequently not been satisfactorily met in many of the wear testing schemes proposed, but is overcome in this method. By finishing lapping with 900-grain crystalon, a polished surface results which shows brilliant fringes under the optical test plane. This is a surface which can be duplicated.

Fig. 5 photographed at fifty magnifications shows the appear-

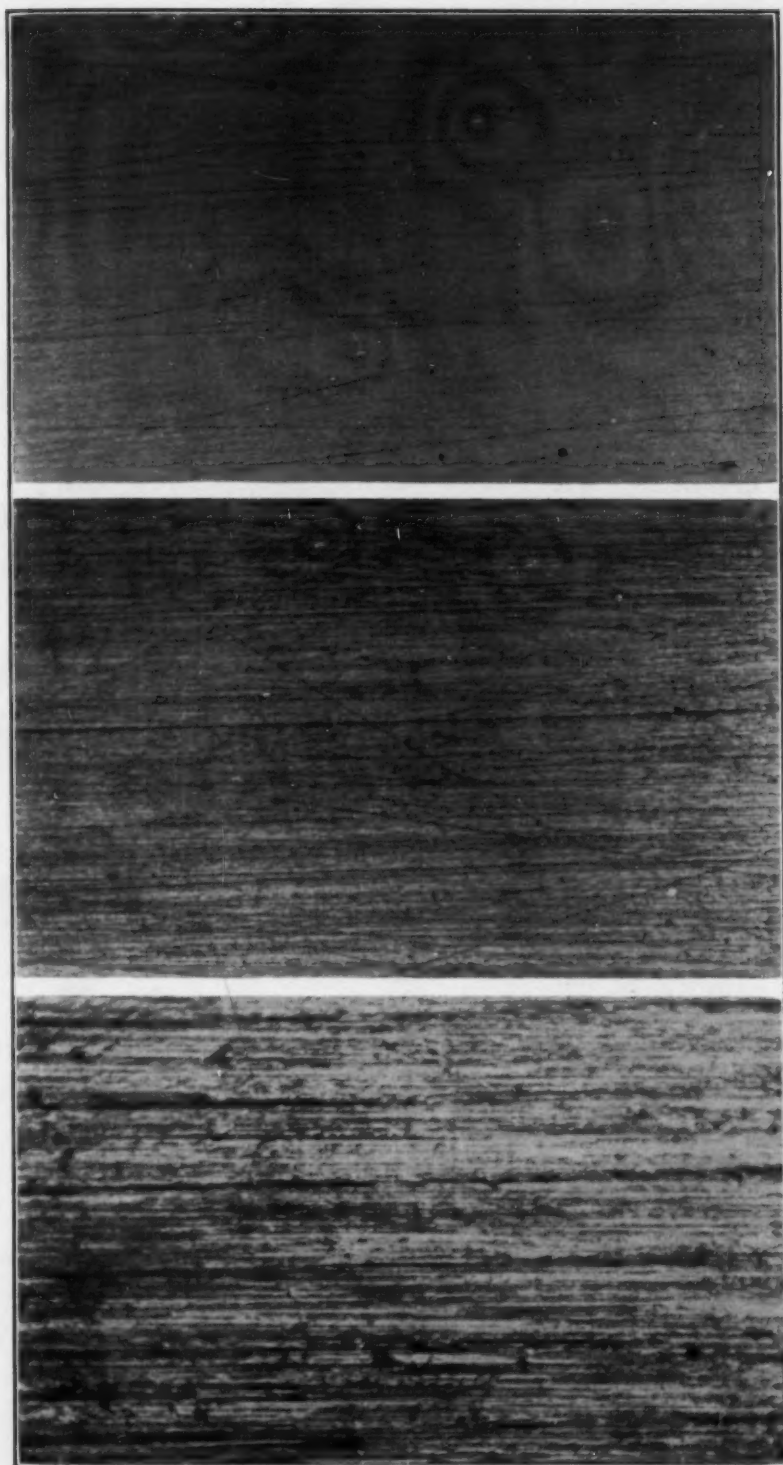


Fig. 5—Microphotos at  $\times 50$  of Finishes on Test Blocks. Top, Finish Obtained by Lapping with 900 Grain Crystolon. Center, Semi-Finished Surface Lapped with 800 Grain Crystolon. Bottom, Original Finish as Ground with 36T22 Aloxite Wheel.



ance of three finishes. On the top is the final finish on a 900-grain lap. The center is the semi-final finish on an 800-grain lap and at the bottom is the ground finish obtained with the 36T22 Aloxite wheel. Soft materials may become charged with abrasive if the surfaces are lapped. It is therefore sometimes necessary to use other classes of finish than the lapped finish. Cast iron, for example, is used with the ground finish which is obtained with the 36T22 Aloxite wheel.

A much clearer conception of the character of these finishes is obtained from the graphs of the surface irregularities taken by a profilograph. This instrument for evaluating surface finishes has been developed by the Timken Roller Bearing Company. We are indebted to W. G. Hildorf of the Timken Roller Bearing Company for profilographs used in this paper.

### *Measurement of Wear*

*Optical Method*—In our work we have used an optically flat, or test plane, as the standard method for measuring the wear of the test blocks.

When an optical test plane is laid on another highly polished flat surface a refraction pattern results. This is best observed by monochromatic light such as an argon, sodium or mercury vapor light. Under these lights the fringes or bands appear in sharp contrast.

We shall not develop in detail the optical considerations which explain the formation of refraction fringes at the interface when a flat polished surface is observed through an optical test plane. This phenomenon is clearly explained by C. G. Peters and H. S. Boyd in the Scientific Paper of the Bureau of Standards No. 436.

It is sufficient to state that the refraction fringes are caused by the interference of light waves through the intervening layer of air at the two faces in contact. Those light waves which are in phase augment each other and the eye observes them as bright bands while those waves which are 180 degrees out of phase cancel each other and darkness results at these wave fronts. Thus a series of bright and dark bands are observed. The distance between the fringes depends on the thickness of the air film between the surfaces. The separation of any two adjacent fringes is one half wave length. For mercury light, which has a wave length of 21.4 microinches, the

separation indicated by two adjacent fringes is 10.7 microinches. A microinch is one millionth of an inch.

If the surfaces being tested are truly flat the pattern observed will be a series of parallel fringes.

The deviation of the fringes from parallelism is a measure of the flatness of the surface being tested. Fig. 6 shows the appearance of a test block under monochromatic light. The photograph at the left shows the parallel fringes. It will be noticed that the fringes curve at the ends. This deviation from parallelism is about two fringes, showing that the edge is two fringes lower than the rest of

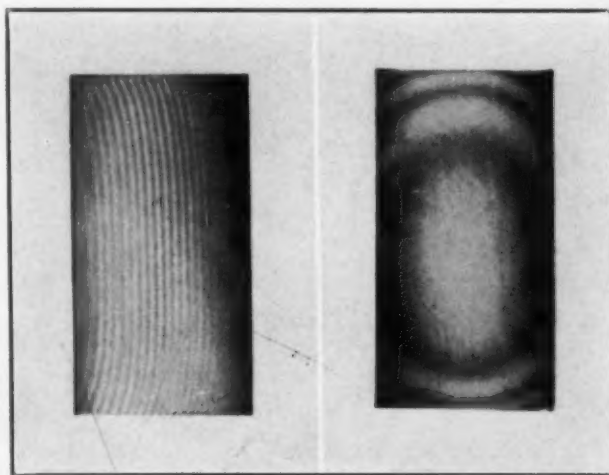


Fig. 6—Lapped Test Block Showing Refraction Patterns. The Pattern on Left Due to Air Film Between Blocks. The Pattern on Right the Test Plane is in Contact with the Test Block.

the block. The deviation from parallelism is usually measured with a Pulfrich instrument.

As the optical test plane is brought into closer contact with the surface being tested, the spacing between the fringes becomes wider. When the two surfaces contact, if the test surfaces are flat, no fringes are formed. This result is shown by the right hand photograph of the same block. The center is in contact and is flat. The ends are not flat by the two fringes which are observed at the ends of the block. A half fringe is seen along the sides. This represents the practical limit of flatness for hand lapping.

Since we do not have a Pulfrich instrument for measuring the deviation, it is easiest to observe the wear when the optical test plane is in contact with the test block. The worn areas are then below the general plane and the resulting pattern is a series of contours. See

Fig. 7. All points on each contour are at the same elevation and each fringe differs by 10.7 microinches. Thus by counting the fringes a measure of the wear in microinches can be obtained. In these tests the wear is usually a trace to three fringes. Seven fringes are the most that can be distinguished on so small a surface. This would be 75 microinches in the center of greatest wear. Fig. 7 shows a nitrided steel block which wore two and one-half fringes when run against a tungsten carbide block which shows no wear. This test was run twenty-four hours at 800 pounds per square inch and one hour at 4000 pounds per square inch.

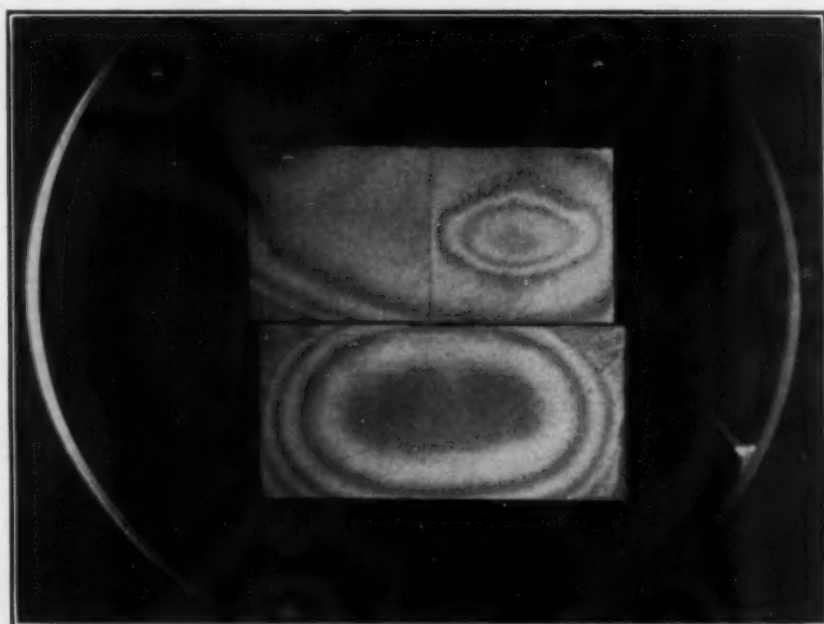


Fig. 7—Nitralloy Block Which Wore Two and One-Half Fringes When Run Against the Carboloy Block Which Shows No Wear.

Of course many couples gall, leaving the surface rough. This holds the test plane away from the surface and the wear cannot be measured. But galling of surfaces indicates that the alloys being tested would not be suitable to use together.

Fig. 8 shows the cabinet used for viewing test blocks. It contains a small mercury vapor tube in the top behind an opal glass. A transformer is in the base. The advantages of this optical method of measuring wear are: The contour of the wear area is shown at the same time as an absolute measure of the wear is obtained. It is easy to record the results by photographing the blocks under an optical test plane. The method is rapid and it is easy to repeat the



measurements and is not expensive to operate. By photographing the three blocks together the results can be recorded for reference and comparison.

Examples of worn surfaces and the refraction pattern they exhibit under an optical test plane are shown in Fig. 9. The upper set is that of a stationary block of Xaloy which shows one-half

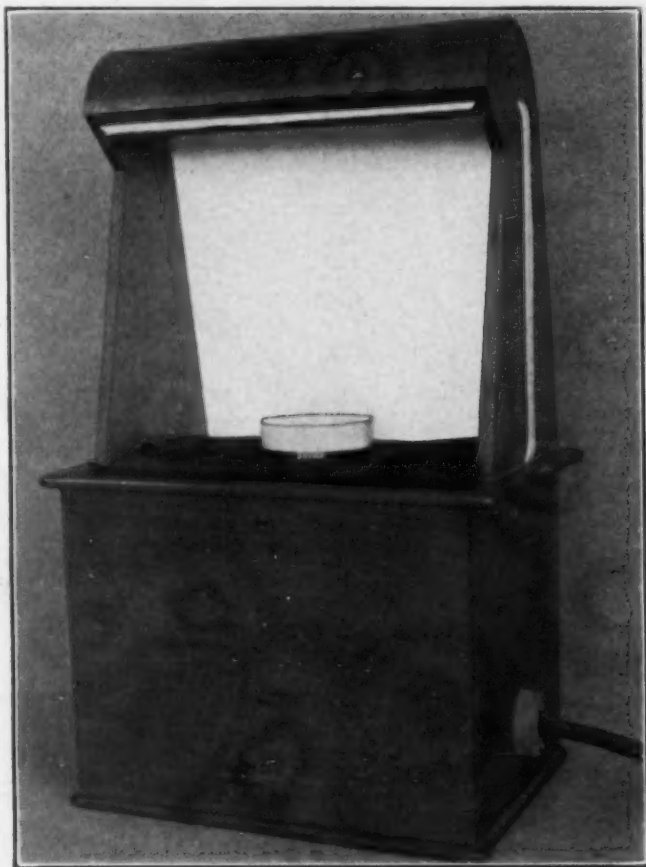


Fig. 8—Mercury Light Cabinet Used For Examining Wear Test Specimens.

fringe of wear when run against a middle block of nitrided steel, which wore one and one-half fringes in twenty-six hours at 1600 pounds per square inch at 122 degrees Fahr. (50 degrees Cent.). Below (Fig. 9) is the complete set of blocks which were run in the original test machine for fifty hours at 200 pounds per square inch and 122 degrees Fahr. (50 degrees Cent.). The center is slightly galled. Wear is tapered from one-half band on the right to one and one-half bands on the left. The center block is nitrided steel. It shows the mutual galling with the stellite 1. The wear is not exactly in the center. It amounts to one-half fringe. The nitrided

steel block on the right ran against the side of the center block not shown. No wear occurred. The bright spot in the center is polish. The dark streaks are discoloration only.

*Electrical Gage*—Although we have used the optical method just

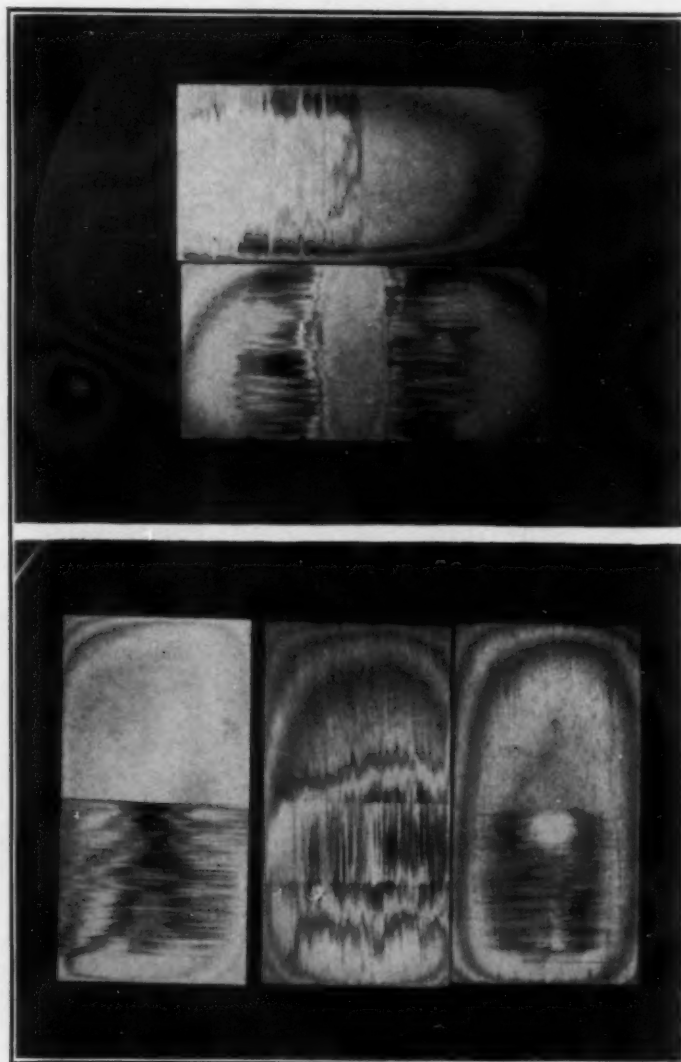


Fig. 9—Above, Xalloy (IR2) Top Which Ran Against Nitralloy. Below, Stellite vs. Nitralloy. Center, vs. Nitralloy. See Text.

described almost exclusively in our work, two other extremely sensitive methods of measuring wear may be used. In cases where a greater amount of wear occurs than can be measured under an optical test plane, a measure of the amount of wear may be obtained by using an electrical gage (Fig. 10) which has been developed in our shop for checking our production. The full scale reading on the

gage represents one thousandth of an inch. This is subdivided into twenty divisions of fifty microinches. Readings can be interpolated to twenty microinches. It is thought that under favorable conditions one interference fringe of ten microinches could be detected with the present gage and the sensitivity in this system of gaging can be increased almost without limit. This method has the advan-

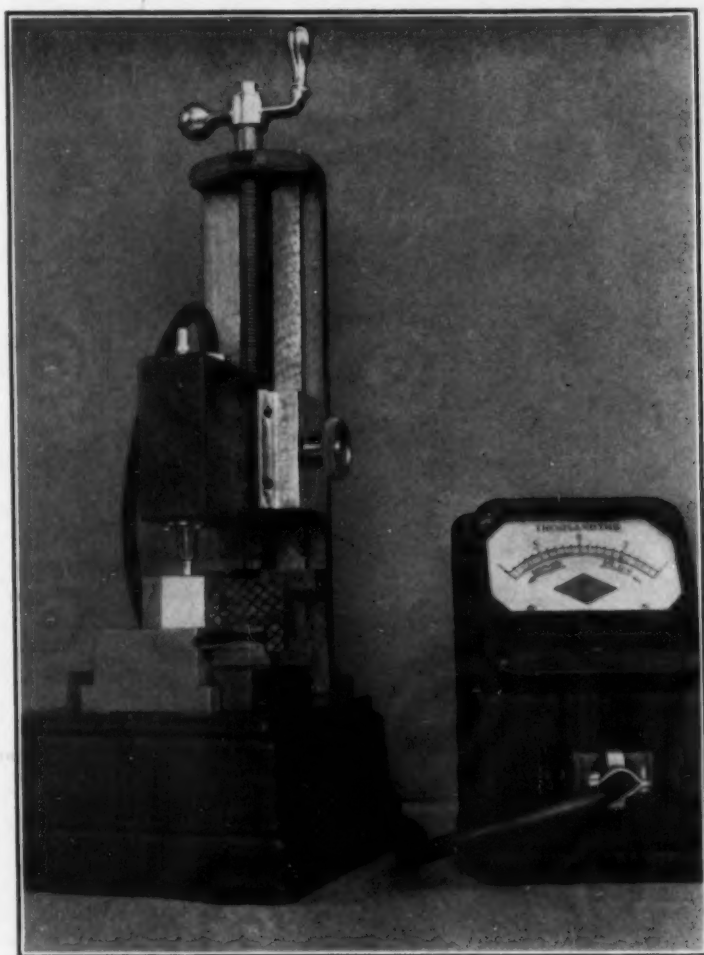


Fig. 10—Electrical Gauge Sensitive to 20 Microinches.

tage of being applicable to cases for which the amount of wear is so great the optical method cannot be used. It serves as a valuable check on other methods. A difficulty with the method is that the two faces of the test block should be parallel to less than one fringe, or a record of the taper can be made before wear testing.

*Profilograph*—The Timken profilograph provides another method of measuring the amount of wear and the character of the worn surface at the same time. Fig. 11 shows three such profilo-



graphs. At the right of the profilograph is the original lapped surface. The trace continues toward the left across the worn surfaces for 0.150 inch. The magnification is 2000 vertically and 30 horizontally. The surfaces of which these are the profiles, were

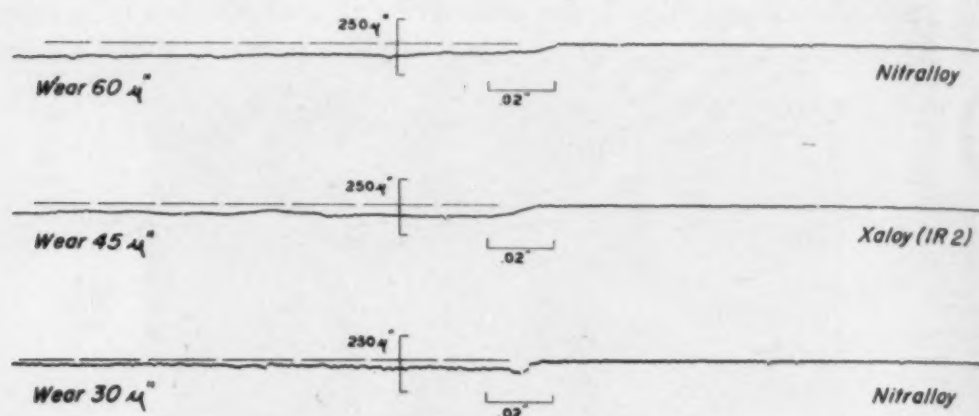


Fig. 11—Profilograph of Worn Surfaces.

chosen to exhibit the characteristics of the original and worn surfaces on Xaloy and Nitralloy. The former tends to wear flat while the latter surfaces usually show slight undulations. The bottom profile was produced when Nitralloy was run against Niresist.

On these profilographs we have drawn a horizontal line indicating the original surface and measured the wear. It amounts to 60, 45 and 30 microinches or about 6,  $4\frac{1}{2}$  and 3 fringes respectively. An advantage of this method of measuring the wear is that the character of the original and the worn surface can be observed. A disadvantage of this method is the time consumed in making the measurements and the lack of the "wear contours" as exhibited by the optical test plane.

#### TEST CONDITIONS

The conditions under which wear tests can be conducted are capable of considerable variation. The conditions chosen for a given test will depend on the information sought. The means of varying the test conditions are discussed in the following sections.

**Lubricants**—Unlike most wear testing which has been carried out on dry, unlubricated surfaces, this method is applicable to lubricated surfaces. Most of our tests have been conducted in crude oil from Santa Fe Springs. This is a 34° A.P.I. crude oil with a viscosity of 38 Saybolt seconds at 50 degrees Cent. Comparison of the wear on reference test blocks tested in other crude oils, from

particular fields, are frequently made. Other series of tests have been run in light refined oils and blends with kerosene. These are described under Results of Wear Tests. A few tests have been conducted with aqueous solutions as lubricants and on dry unlubricated surfaces in air.

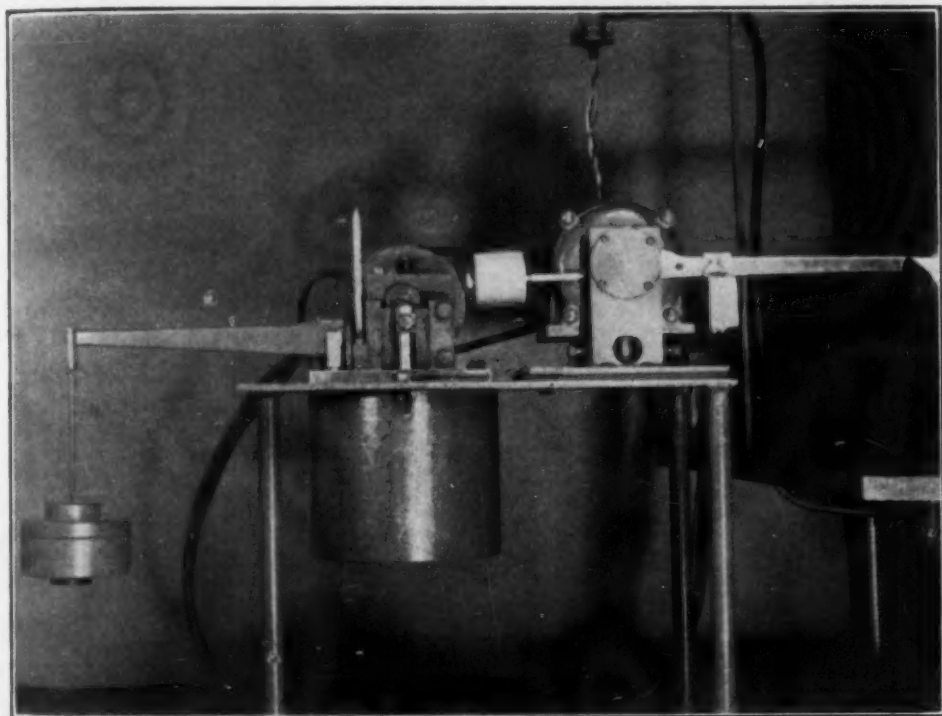


Fig. 12—Test Machine in Operation Position.

*Pressures*—By varying the weights hung on the lever arm, the pressure on the test blocks can be varied. Increasing the weight on the arm by one-half pound increases the pressure by 100 pounds per square inch. The pressures used in these tests have ranged from 13 pounds per square inch to 4000 pounds per square inch. The most frequently used load is 1600 pounds per square inch.

*Temperature*—The temperature of the oil being pumped at 8000 feet at Santa Fe Springs is about 220 degrees Fahr. It was impractical to hold the oil at this high temperature because our testing machine is not gas tight. The light fractions in the crude oil evaporated through the fits at this high temperature and the oil rapidly changed in viscosity. We standardized at 122 degrees Fahr. and most tests were run at this temperature. An immersion heater, stirrer and thermostat provide the temperature control.

*Time*—The duration of the runs was intended to average about

twenty-four hours. The actual time varied considerably. Galling has been the chief cause for stopping a test prematurely. Some tests have been continued for seven days.

*Dynamometer*—Recently a dynamometer has been added to the testing machine. This is shown in Fig. 12. The purpose of this dynamometer is to measure the frictional characteristics of different

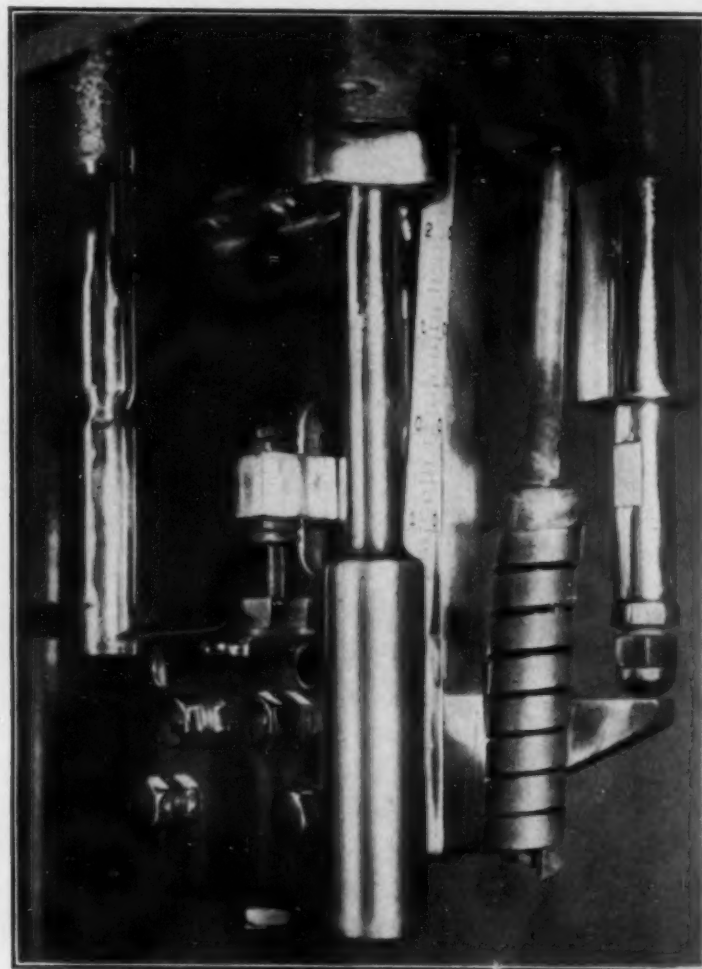


Fig. 13—A Close View of the Back Showing Emulsifying Pump, Stirrer, Heater Thermostat and Thermometer.

metallic couples when run in the same oil and the friction of various oils on the same couples. Too few tests have been made on this basis to report any conclusions as related to wear.

*Emulsifying Pump*—Some oil wells produce considerable water. When this is emulsified in the crude oil it is sometime difficult to separate. At high pressures this water and emulsion is very destructive to the pumps. It causes cutting and corrosion.



We wished to study these effects so an emulsifying pump has been added. Fig. 13 shows this plunger pump. It is on the back of the sample holder. The plunger is actuated by the reciprocating sample holder. It has a ball check intake valve and exhausts through a 0.013-inch diameter orifice to promote emulsification. The stirrer which is gear driven off the countershaft assists in maintaining any suspensions being used. Wear under these conditions is usually excessive.

*Reference Conditions*—While it is not possible to standardize on one set of conditions due to the varied objectives of our tests, the most frequently used conditions have been: Alloys tested at 1600 pounds per square inch at 122 degrees Fahr. in Santa Fe Springs crude oil without preliminary "breaking in." As a reference for wear resistance repeated tests under these conditions show nitrided Nitralloy will wear about two fringes and Xaloy (IR2)\* about one and one-half fringes in twenty-four hours.

If a test shows that a block wears one fringe or 10.7 microinches per hour, that is equivalent to 0.025 inch in one hundred days. If the rate of wear is 10.7 microinches in twenty-four hours the wear in one hundred days would be 0.001 inch. The pumps run continuously and the wear on the parts averages much less than 0.001 inch per one hundred days. No alloy which will not run under these conditions can be considered to have outstanding resistance to wear for our purpose.

*Breaking In*—It is considered that the rate of wear in these tests is greatest at the start tending to decrease as the asperities on both surfaces are rubbed off. Gradual loading as polish develops certainly increases the chances of running successfully and decreases the probability of scoring or galling. However, the pumps must be put on full load immediately without benefit of previous "breaking in." As a consequence alloys which require breaking in are not favored.

#### RESULTS OF WEAR TESTS

This wear testing has been conducted for four and a half years. Although over three hundred tests have been run in this machine, the work is far from complete and we regard this summary of test results as a progress report. About fifty different metals and non-metals have been tested. The number of combinations has been

\*Xaloy (IR2) is a white cast iron boride containing 4 per cent nickel. It is centrifugally cast in tubes for liners.

**Table I**  
**List of Some Materials Tested and Rockwell Hardness**

	15N	30N	30T	A	B	C
Nicholson Test File (For Reference) <sup>1</sup> .....	93.7	85	..	86	..	67
Nitralloy—Average Hardness .....	95	86	..	..	..	..
Hardest .....	95.5	88	..	..	..	..
Low Hardness .....	94.5	85.5	..	..	..	..
Xaloy (IR1) .....	..	75	..	81	..	56
Xaloy (IR2) .....	..	82.5	..	86	..	66
Xaloy (IR3) .....	..	82	..	86	..	66
Xaloy (IRC) .....	..	81	..	84	..	64.5
Xaloy (IR45%W) .....	..	86	..	88	..	..
Cast Iron Average .....	..	35	76	57	94	13
Cast Iron Heat Treated .....	..	74	..	79.5	..	56
Ni-Resist Sand Cast .....	..	..	66	50	76	..
Ni-Resist 4 to 6% Cr. Sand Cast .....	..	..	85	67	105	29
Nitralloy Cast Iron (Nitrided) .....	..	84	..	83	..	..
Cast Iron Meehanite, Heat Treated .....	..	..	..	82	..	57
Cast Iron Meehanite .....	..	..	..	62	..	20
SAE 4615 Fine Grain Quenched from Pot in Oil (Ground) .....	..	81	..	85.5	..	66
5% Chromium Steel (Nitrided) .....	..	87	..	85	..	..
Chromovan Tool Steel (Nitrided) .....	..	87	..	85	..	64
High Speed Steel W18 Cr. 4 V1 (Soft) .....	..	80.5	..	83.5	..	62
High Speed Steel W18 Cr. 4 V1 (Nitrided) <sup>2</sup> .....	96	89	..	89.5	..	69
High Speed Steel W19 Cr. 4 V2 Co. 7 Mo 1..	..	80	..	83	..	63
High Speed Steel W19 Cr. 4 V2 Co. 7 Mo 1 Nitrided .....	96	89.5	..	87	..	65
High Speed Steel W19 Cr. 4 V1 Co. 5 (Soft)	..	77	..	81.5	..	59.5
Stainless Steel Type 440 (H17) .....	..	76.5	..	82	..	58
Chrome Plate on Hardened SAE 4615 <sup>3</sup> .....	92.5	82.5	..	80	..	..
Cold Rolled Chapmanized <sup>4</sup> .....	..	71	..	..	..	..
Premet Aged .....	..	83	..	85.5	..	66.5
Union Carbide Steel 29.7% Cr. 2.5% Cr. ....	..	80	..	83.5	..	63

	15N	30N	30T	A	C	F
Stellite 1 Overlay (Average) .....	..	72	..	80	55	..
Stellite 2A .....	..	85	..	87.5	69	..
Stellite 3 .....	..	78.5	..	83.5	61	..
Stellite 5 Overlay .....	..	73.5	..	79	..	..
Stellite 6 .....	..	65.5	..	75	46	..
Stellite 6 Overlay .....	..	67.5	..	76	..	..
Stellite 12 Overlay .....	..	70	..	76	..	..
Stellite 13 .....	..	84	..	87.5	..	..
Stellite 13 Softer .....	..	82	..	85	..	..
Stellite J .....	..	78.5	..	83.5	61	..
Jadson Seat Metal Overlay .....	..	42	..	61.5	20	..
Cristite Overlay .....	..	67.5	..	76	48	..
Cast Tungsten Carbide .....	97	93	..	..	..	..
Carboloy Grade 44-A .....	95	89	..	..	..	..
Cast 12½% Co. WC Alloy .....	92	86.5	..	..	..	..
Cast Colmonoy Cr-Boride Crystals .....	95	87	..	..	..	..
Cast Colmonoy No. 3 .....	..	82	..	86	65	..
Cast Colmonoy No. 6 .....	..	75	..	80.5	58	..
K. Monel Cold Drawn .....	..	51	..	66.5	31	..
K. Monel Aged .....	..	60.5	..	72	41	..
Sintered Alloy 43% Co. Bal. WC .....	..	73	..	80	55	..
Sintered Alloy 40% Co. Bal. WC .....	..	75	..	81	57	..
Bakelite No. 6240 .....	..	..	74	..	..	63
Bakelite No. 6031 .....	..	..	70	..	..	60
Micarta (Air Brake) .....	..	..	79	..	..	72
Micarta (Tail Bearing) .....	..	..	66	..	..	68
Micarta 238; 285; T31516 .....	..	..	67	..	..	50
Micarta 281; T3256Y .....	..	..	72	..	..	60
Micarta 223 .....	..	..	68	..	..	52
Graphite Electrode 139 .....15T....14	..	..	..	..	..	..

<sup>1</sup>The hardness of the body of a Nicholson Test File is given for reference.

<sup>2</sup>Note the high hardness of Nitrided High Speed Steel.

<sup>3</sup>Hardness readings on heavy hard chromium plates up to 0.025 inches in thickness have repeatedly been lower than its apparent hardness. It cannot be filed.

<sup>4</sup>Chapmanized by Chapman Valve Manufacturing Company, Indian Orchard, Mass.

much greater. The test conditions have been varied as demanded by the information sought. Materials varying in hardness from cast tungsten carbide to micarta and graphite have been used.

We have been able to determine the suitability of an alloy for use in service by determining its behavior in combinations which will run against other alloys and to eliminate combinations which will not run together. It probably should be stated that it has been largely a case of elimination, for many of the alloys we hopefully tried we have found not to run successfully in combinations which we desired to use. As has been stated, nitrided Nitalloy and Xaloy (IR2) have established themselves as reference standards in our work. This is because of their great resistance to wear and the ability of Nitalloy to run against itself under extreme pressure. It is well known that great hardness alone is no criterion of wear resistance. However, the requirements to be met, limits the choice of alloys to those of high strength and unusual hardness. It is on this class metals that most of our testing has been done. We believe that the method can be used for testing softer metals such as bearing metals. This necessitates developing different methods of preparing standard wear surfaces as it is thought that a standard finish for softer alloys could be developed by closely controlled broaching. We are currently engaged in testing the wear resistance of some synthetic plastics for service in corrosive wells where the pressures are not high.

While most of the alloys tested were unusually hard, many did not offer wear resistance in the sense that they galled. Table I is a list of some of the alloys and nonmetallics tested to date. Their Rockwell hardness in the most appropriate scales are listed and are the values obtained on new superficial and standard Rockwell hardness testers. The testing was carefully done, all at one time. The hardnesses given are all observed values. The machines were repeatedly checked against standard high range test blocks as the testing progressed. Most of the tests were taken on wear test blocks which were lapped flat on both sides and finished with 900-grain crystolon. On the more homogenous alloys, check readings were readily obtained and the agreement among the different scales and with the Rockwell conversion sheet is good. Where agreement is not so precise, it is felt that this is due to the characteristics of the particular alloy, since the readings for one of the Rockwell ranges usually checked the conversion sheet.

B	C
..	67
..	..
..	..
..	56
..	66
..	66
..	64.5
94	13
..	56
76	..
05	29
..	..
..	57
..	20
..	66
..	..
..	64
..	62
..	69
..	63
..	65
..	59.5
..	58
..	..
..	66.5
..	63

C	F
55	..
69	..
61	..
..	..
46	..
..	..
..	..
..	..
61	..
20	..
48	..
..	..
..	..
..	..
65	..
58	..
31	..
41	..
55	..
57	..
..	63
..	60
..	72
..	68
..	50
..	60
..	52

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## SOME UNUSUAL ASPECTS OF MALLEABLE IRON MELTING

BY ALFRED L. BOEGEHOLD

### *Abstract*

*It is shown that the sum of carbon and silicon permissible for obtaining white fracture in a certain size section varies depending upon melting conditions. The sum of carbon plus silicon may vary from as low as a maximum of 3.20 per cent for induction furnace melting to as high as 4.20 per cent maximum for cupola electric furnace melting to produce white fracture in a 2-inch round casting. The important steps in experiments to discover causes for this variation are given. The most important causes discovered are the carbide stabilizing influence of moisture either in contact with the metal while melting or after melting and the graphitizing influence of iron oxide additions. The influence of iron oxide upon annealing is discussed showing that it retards annealing principally through its effect upon the location of temper carbon spot formation. The effect of copper with and without oxide present upon temper carbon distribution is shown to be such that it is indicated that the retarded annealing caused by iron oxide additions is solely the result of the increased distance between temper carbon spots, for when this is prevented by copper additions the annealing is not retarded. It is proposed that carbide stabilizers and graphitizers be more critically defined or grouped according to their respective influences during solidification and during annealing.*

**Y**OUR speaker greatly appreciates the privilege of honoring and perpetuating the memory of Edward de Mille Campbell in this the Thirteenth Campbell Memorial Lecture.

Professor Campbell was a leader of metallurgical investigation and thought in the first quarter of this century. One of his favorite investigations pertained to the composition of iron carbide. It was in an experiment concerning this question that Professor Campbell lost his eyesight.

This is the Thirteenth Edward DeMille Campbell Memorial Lecture, presented by Alfred L. Boegehold, head of metallurgical department, Research Laboratories Section, General Motors Corp., Detroit. The lecture was presented at the Twentieth Annual Convention of the Society, held in Detroit, October 17 to 21, 1938.

Like Professor Campbell, we are interested in iron carbide—not its composition but how it behaves in hard iron used for making malleable iron.

Malleable iron was selected as a subject for this lecture partly because of its prominence in the automotive industry, partly because we believe this material should receive more attention than it has in the past, but principally because we were investigating malleable iron when this lecture had to be written.

A large portion of the investigations reported in this lecture were conducted in the laboratory with the help of W. E. Jominy and R. H. Terry, with whom many hours were spent in planning and discussing experiments. Carl Joseph, chief metallurgist of the Saginaw Malleable Iron Co., supplied the pyramidal castings and other castings made from production iron studied in this investigation.

Fifteen years ago most malleable iron was melted in air furnaces. For the general run of automotive castings it was necessary to hold the carbon not over 2.60 per cent and the silicon not over 1.00 per cent in order to obtain a white fracture in all castings. Even when composition was held within this range, fracture tests had to be made on every heat to be sure that the iron showed a fracture sufficiently free from primary graphite to make good malleable iron after annealing. Without knowing why some heats of the proper composition showed too much or too little tendency to form primary graphite in the form of mottles, the metallurgist could always make the necessary adjustments in composition to get the desired fracture. When the duplex process for making malleable iron was instituted involving melting in a cupola, followed by heating to casting temperature in a direct arc electric furnace, instead of a limiting total carbon plus silicon of 3.60 per cent to which air furnace iron was confined, the duplexed iron could contain 2.75 per cent carbon and 1.25 per cent silicon, or a total of 4.00 per cent carbon plus silicon. In some cases duplexed iron has contained as high as 2.90 per cent carbon and 1.30 per cent silicon without too much mottling occurring in a 1½-inch diameter sprue. The upper limit for carbon and silicon in duplexed iron which will insure freedom from mottling will fluctuate in the same way as in the case of iron melted in the air furnace except that the range of fluctuation is at a higher level. The reason for this fluctuation is no better understood than the similar fluctuation in air furnace malleable iron. The empirical method of control by

breaking test sprues for examination serves, as in air furnace melting, to circumvent our lack of knowledge as to the cause of this variation. The ability to use higher percentages of carbon and silicon in the duplex process is attributed to the use of larger quantities of steel in the charge and to heating the iron hotter in the electric furnace. This does not explain however the variable carbide retaining power with amount of steel in the charge and superheating temperature constant. The magnitude of the variation in carbide retaining power of hard

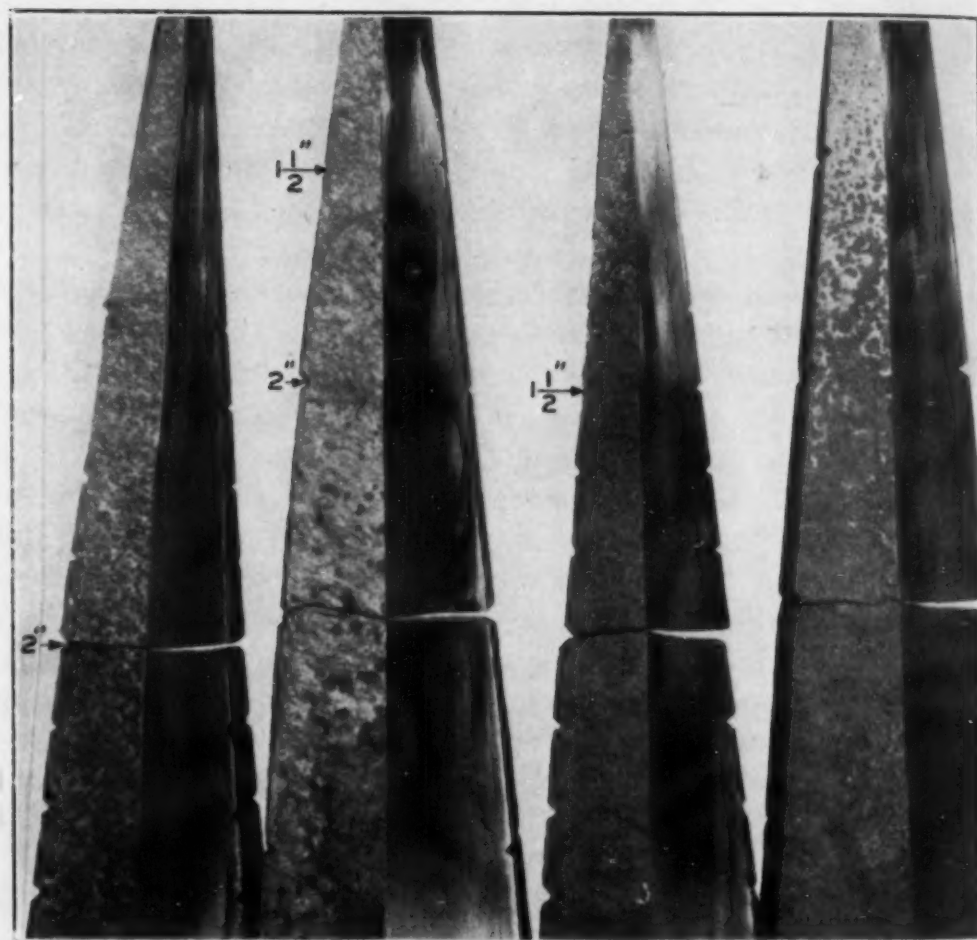


Fig. 1

2 Hours After Melting Started	
Cupola Iron	Electric Furnace Iron
2.79% C	2.71% C
1.11% Si	1.16% Si
0.37% Mn	0.37% Mn
0.11% S	0.11% S

9 Hours After Melting Started	
Cupola Iron	Electric Furnace Iron
2.80% C	2.75% C
1.25% Si	1.18% Si
0.41% Mn	0.40% Mn
0.11% S	0.11% S

iron as regularly made in production is illustrated in Fig. 1. This shows fractured pyramid castings displaying the occurrence of white fracture relative to section size. Castings were made from both



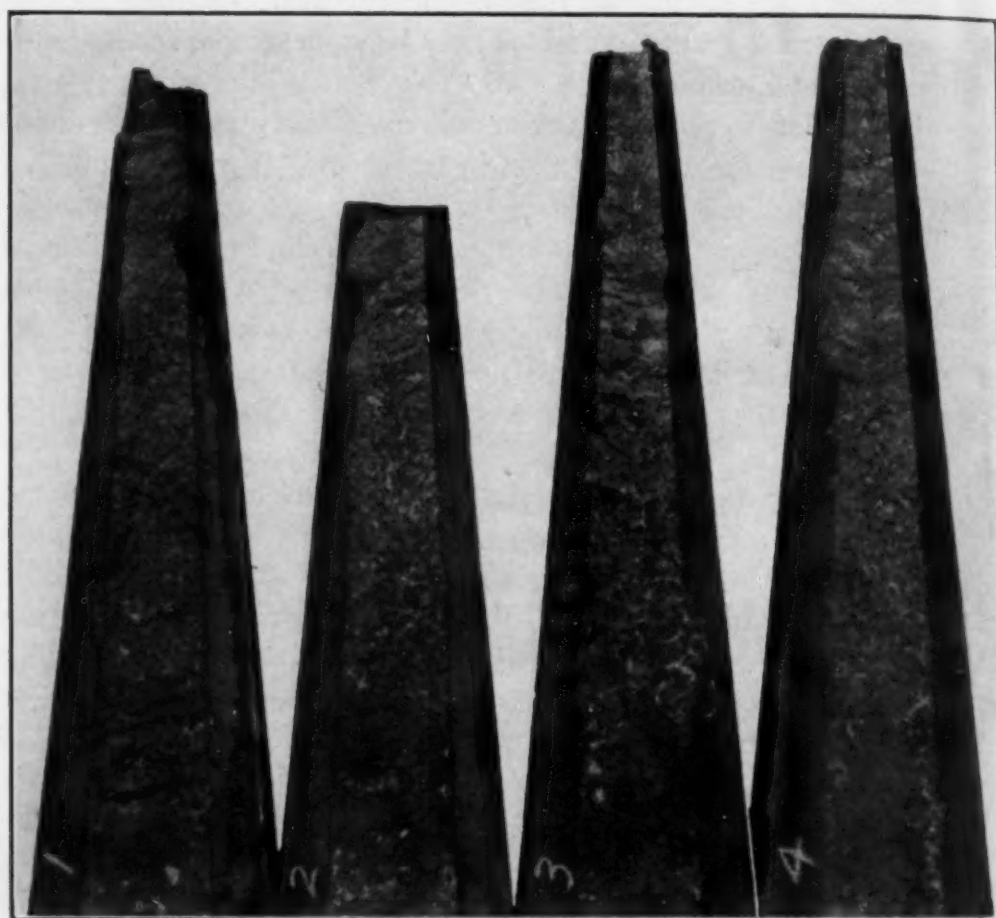
cupola iron and from iron after heating to casting temperature in the direct arc electric furnace. These four fractured castings disclose several interesting facts.

1. The cupola iron shows considerably less carbide retention than the iron after heating in the electric furnace. Castings of two sizes were used but comparison may be made at like sections, for example at 2 inches.
2. The mottled structure in the cupola metal consists of gray areas of much smaller diameter than in the electric furnace metal.
3. The castings made 7 hours after the first two were cast show decidedly less carbide retaining power than the first two although they are quite similar in composition.
4. The reduced carbide retaining ability in the second pair of castings is similar in both cupola metal and electric furnace metal—suggesting the origin of the influence in the cupola melting process.
5. The electric furnace iron cast 9 hours after melting started shows some of the small diameter mottles which characterize the cupola metal.

Although the carbide retention in the second electric furnace casting was toward the low side, it still had sufficient freedom from mottling in the 1½-inch section to be satisfactory for making the regular run of production castings. The magnitude of this variation in carbide retaining ability had not formerly been realized because fracture tests were always made on a sprue 1⅝-inch in diameter at the midpoint. The pyramid castings fractured longitudinally give much more extensive information. Even with the realization of the extensive fluctuation in carbide retentivity, there was no urgent demand for a discovery of the causes because, control was simply and easily accomplished by means of adjustments made depending upon the appearance of fractured sprue test bars. It seems evident that a faster annealing iron would result if the causes for increasing carbide retentivity with high silicon content were better understood, therefore, it is somewhat surprising that so little work has been done on this problem. It was not until we experienced failure in obtaining white iron in a laboratory melt that was similar in composition to the production malleable iron that we were sufficiently impressed as to the importance of knowing the reason for this decrease in carbide retentivity.

Our experiment consisted of an attempt to make some alloy

malleable iron in a high frequency induction furnace with a composition which by comparison with the production iron should have been white in a 2-inch round section. The fracture of this iron in a 2-inch round, however, was not white nor even speckled, but all gray. The



Heated to 2600° F. 2700° F. 2800° F. 2900° F.  
Cupola—Electric Furnace—Production Hard Iron

same result was obtained when a melt was made with the same composition as the production metal. This experience convinced us that our knowledge as to principles governing carbide retentivity in iron was sadly lacking. Without being able to duplicate production results in laboratory melts, it would be impossible to develop alloys in the laboratory that could be used in production. It became obvious, therefore, that these principles would have to be discovered before proceeding with the development of alloy malleable iron. The fact that increasing the temperature of the molten iron has a strong carbide retaining effect has been long known. In Fig. 2 are shown

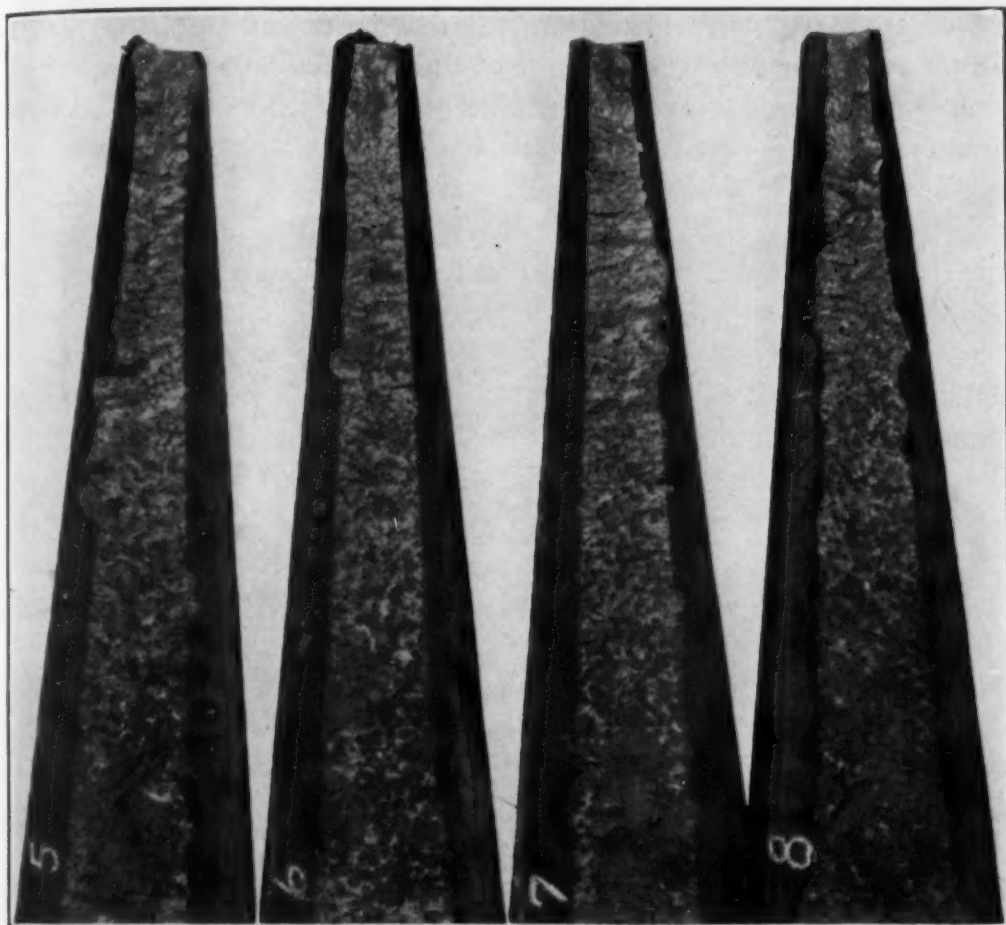


Fig. 2

2900° F.—5 Min.	2900° F.—5 Min.	2900° F.—5 Min.	2900° F.—5 Min.
Cast at 2900° F.	Cast at 2800° F.	Cast at 2700° F.	Cast at 2600° F.
Cupola—Electric Furnace—Production Hard Iron			

fractured wedge castings made from production iron showing the extent of white and mottled fracture as a result of various cycles of heating and cooling of the molten iron.<sup>1</sup> At the 2-inch section, the fracture of the iron heated to 2600 degrees Fahr. (1425 degrees Cent.) is about 60 per cent white and when heated to 2700 degrees Fahr. (1480 degrees Cent.) is about 75 per cent white at that section, consequently our task was to produce iron of similar composition and fracture without exceeding a melting temperature of 2700 degrees Fahr. Although heating to 2800 and 2900 degrees Fahr. (1535 and 1595 degrees Cent.) increases the carbide retentivity, our plan consisted of ascertaining the way to obtain white fracture without taking advantage of the carbide stabilizing effect of superheating.

The method of attack consisted of making a large number of

<sup>1</sup>Carl F. Joseph, "Measuring and Controlling Pouring Temperatures and Fluidity." *Transactions, American Foundrymen's Association*, 1936.



exploratory melts to which additions were made and treatments given in an endeavor to obtain some clues as to measures that would increase carbide retentivity. Naturally, the things that were tried were those that have been reported to have a carbide stabilizing effect. Since the high frequency induction furnace is a common melting unit for laboratory investigations, it seems appropriate to include here a number of the melts which were made before it was found out what the important controlling factors were, just to illustrate how contradictory the results can be and so that the information will be available to help those workers who are trying to find new alloys by induction furnace melting. Part of this paper then becomes a partial case history of an attempt to make white iron in an induction furnace of the same composition as employed in the cupola-electric furnace production method of melting.

The melting stock used after the first few heats consisted of small white iron castings, enough of which from one day's production cast were obtained at the beginning so that the same melting stock could be used for all tests. Melting down time was controlled nearly constant by using a constant power input. Additions of ferromanganese, ferrosilicon and wash metal to adjust the composition were always made in the same order. Temperature readings with a Leeds and Northrup optical pyrometer were corrected for oxide film on the surface of the metal when present. When gas was bubbled through the melt it was introduced through a quartz tube first wrapped with a double winding of asbestos cord, smeared with a high temperature cement, and then rolled in powdered magnesia. This sort of tube stood up well at temperatures as high as 2950 degrees Fahr. (1620 degrees Cent.). The total charge melted in these heats was 25.4 pounds.

The first heat which was intended to duplicate the composition of commercial iron contained 2.76 per cent carbon and 1.24 per cent silicon. This was made from steel scrap, ferrosilicon, and carbon added in the form of graphite. The metal was melted in air, heated to 2650 degrees Fahr. (1455 degrees Cent.), poured into a plumbago crucible which had been heated to 1700 degrees Fahr. (925 degrees Cent.), so that any graphite at the surface was burned off, and cast into a green sand mold for a 2-inch round casting 8 inches long. The fracture of this casting pictured in Fig. 3, M-33, was completely gray. The use of graphite to supply the carbon was held responsible for the gray fracture, consequently the next heat was made with the carbon

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added in the form of wash metal containing 3.86 per cent carbon, all in combined form. In addition to this, because of the carbide stabilizing influence of hydrogen reported by Boyles,<sup>2</sup> the melting

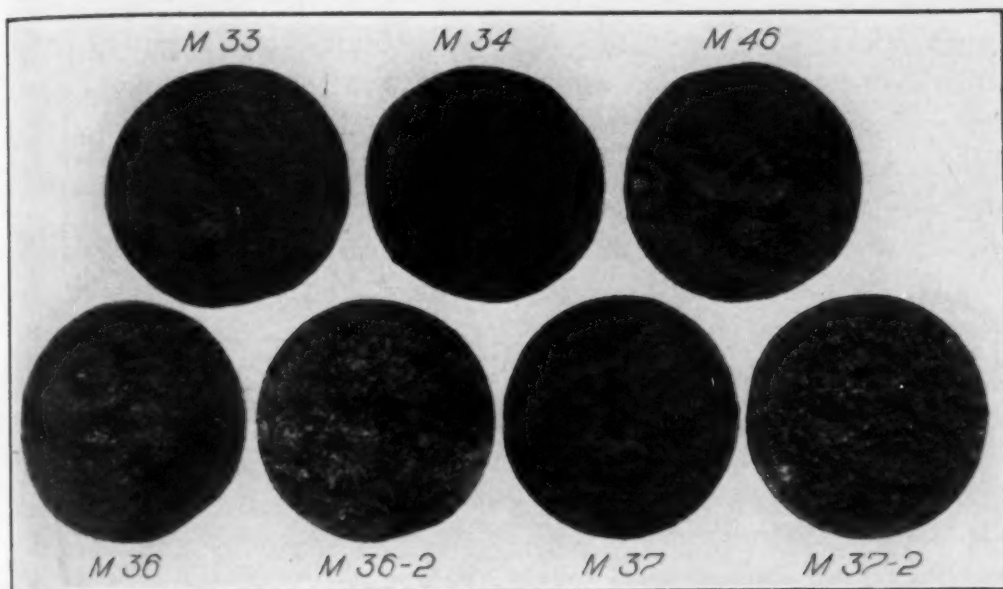


Fig. 3

M33—2.76% C, 1.24% Si, 0.40% Mn. Heated to 2650° F. Carbon in Charge Added as Graphite. 0.8 Gr. Water/Cu. Ft. Air. M34—2.49% C, 1.19% Si, 0.39% Mn. Heated to 2640° F. Carbon Added as Wash Metal. Dry Hydrogen in Furnace While Melting. 1 Gr. Water/Cu. Ft. Air. M46—2.78% C, 0.89% Si, 0.37% Mn. Heated to 2660° F. Made from Shrink Bobs, Steel and Graphite. Poled 2 Min. with Green Sticks. 2.5 Gr. Water/Cu. Ft. Air.

M36—2.42% C, 1.10% Si, 0.35% Mn. Heated to 2660° F. Made from White Iron Castings and Wash Metal. 1.3 Gr. Water/Cu. Ft. Air. M36-2—2.40% C, 1.00% Si, 0.35% Mn. Heated to 2660° F. M36 Plus 5% Steel. M37—2.31% C, 1.17% Si, 0.39% Mn. Heated to 2660° F. Dry H<sub>2</sub> in Furnace 1.3 Gr. Water/Cu. Ft. Air. M37-2—2.32% C, 1.10% Si. Heated to 2680° F. M37 Plus 5% Steel.

crucible during heating and melting was supplied with dry hydrogen. The resulting composition was 2.49 per cent carbon and 1.19 per cent silicon, or a total carbon plus silicon of 3.68 per cent. This metal heated to 2640 degrees Fahr. (1450 degrees Cent.) and cast also produced a completely gray fracture in a 2-inch round, as shown in Fig. 3, M-34.

The failure of the hydrogen to produce a white fracture was assumed to be on account of not heating to a high enough temperature in accordance with Boyle's findings. This still left us with no evidence as to why gray fracture occurred with such low carbon and silicon.

Another fracture of casting M-46 is shown in Fig. 3 made from a melt using shrink bobs from white iron castings, steel, and graphite

<sup>2</sup>Alfred Boyles, *Transactions, American Institute of Mining and Metallurgical Engineers*, Vol. 125, Iron and Steel Division, 1937, p. 141.

in the charge. The silicon content of this heat was purposely reduced considerably in order to see if a white fracture could be obtained by this means. After melting and heating to 2660 degrees Fahr. (1460 degrees Cent.), the molten metal was poled for two minutes with green sticks. This measure was used because it is reputed to be capable of increasing the carbide retentivity of metal of this composition. Again the fracture of the 2-inch round casting was completely gray. Again there is the use of graphite in the charge to suspect as the cause for the gray fracture.

In order to eliminate the possibility of influence of free graphite from any source whatever, a heat was made entirely from white iron castings and wash metal. This was heated to 2660 degrees Fahr. (1460 degrees Cent.) and even with 2.42 per cent carbon and 1.10 per cent silicon, with a total of only 3.53 per cent carbon plus silicon, the fracture was completely gray. See M-36, Fig. 3. To this metal after the first casting was poured was added 5 per cent steel as it is generally considered that the addition of steel tends to increase carbide retentivity to a greater extent than would be caused by the consequent reduction of carbon and silicon. The result of this addition, shown in Fig. 3, M-36-2, did not support this reputation for steel additions although a small amount of white was present in the fracture of casting made from this metal which contained 2.40 per cent carbon and 1.00 per cent silicon, or a total of 3.40 per cent carbon plus silicon. This is far below the amount of carbon and silicon present in commercial duplexed white iron where the carbon runs about 2.75 per cent and the silicon about 1.25 per cent, or a total of 4.00 per cent carbon plus silicon. It is obvious then that some unknown strong graphitizing influence is operating to prevent white fracture in the castings made from these experimental heats.

Heat M-37 was made like M-36 but with dry hydrogen introduced into the furnace during melting. This heat contained lower carbon and silicon than the previous one but at 3.48 per cent total carbon plus silicon the dry hydrogen did not produce a white fracture. Even after 5.00 per cent steel was added in M-37-2, reducing the total carbon plus silicon to 3.42 per cent, was there any but a slight indication of carbide appearing in the fracture.

Since there has been evidence in production and in the literature that moisture in contact with molten iron causes an increase in carbide retentivity, an experiment was made to see whether it would have that effect when melting in the high frequency induction fur-



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nace. A heat was made from white iron castings and wash metal and a test bar cast prior to treatment with moisture. In all subsequent heats, unless otherwise mentioned, the charge used will be wash metal and small white iron castings made by the cupola electric

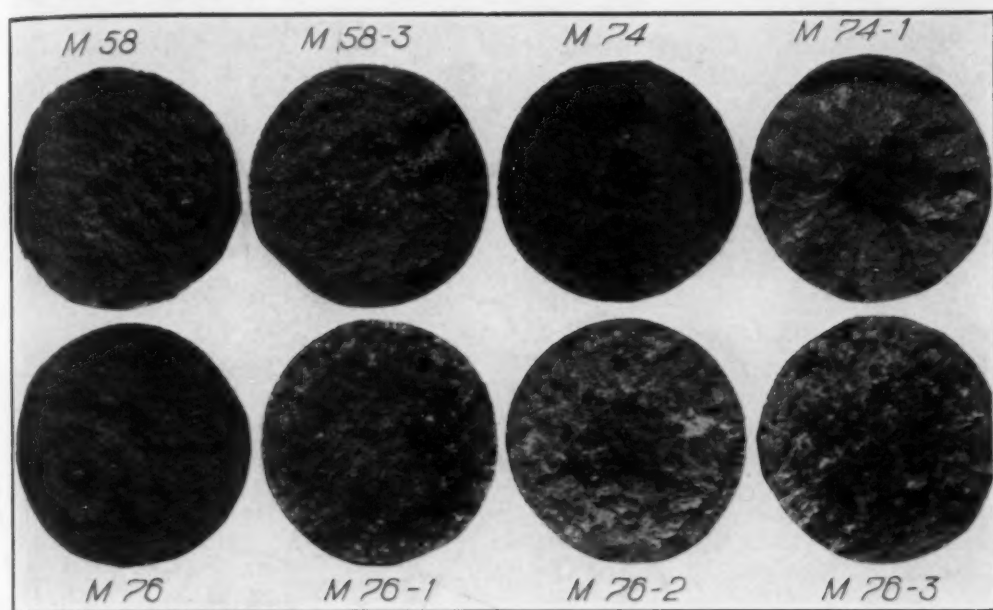


Fig. 4

M58—2.61% C, 1.26% Si, 0.54% Mn. Heated to 2640° F. 1.4 Gr. Water Per Cu. Ft. Air. M58-3—2.48% C, 1.17% Si, 0.43% Mn. Heated to 2715° F. Moist Air Passed Through Melt for 8 Min. M74—2.65% C, 1.12% Si, 0.36% Mn. Heated to 2640° F. 3.6 Gr. Water Per Cu. Ft. Air. No Transfer Ladle. M74-1—2.72% C, 1.07% Si, 0.34% Mn. Heated to 2715° F. Steam Passed Through Melt for 4 Min.

M76—2.72% C, 1.17% Si, 0.39% Mn. Heated to 2475° F. 3.2 Gr. Water Per Cu. Ft. Air. M76-1—2.65% C, 1.14% Si, 0.40% Mn. Heated to 2640° F. M76-2—2.53% C, 1.12% Si, 0.42% Mn. Heated to 2850° F. M76-3—2.53% C, 1.12% Si, 0.40% Mn. Heated to 2850° F. Cooled to 2475° F.

furnace process in order to be sure that no primary graphite is present to promote a gray fracture. The composition of this heat was 2.61 per cent carbon and 1.26 per cent silicon and the fracture shown at M-58, Fig. 4, is again gray in a 2-inch round. After passing moist air through the melt for 8 minutes at about 12 cubic feet an hour, in which time the composition changed to 2.48 per cent carbon and 1.17 per cent silicon, the fracture was still gray. The moist air was produced by passing compressed air through water at a temperature of 160 to 180 degrees Fahr. Here again then we get a contradictory result. A similar experiment was then performed except that steam was passed through the melt instead of moist air and here again the iron melted and heated to 2640 degrees Fahr. (1450 degrees Cent.) with a total carbon plus silicon of 3.77 per cent and cast into a test bar prior to the steam treatment was again gray

but after passing steam through the melt for 4 minutes, the fracture in a 2-inch round was completely white with a total carbon plus silicon of 3.79 per cent. M-74 and M-74-1, Fig. 4. Here was the first evidence supporting the idea that moisture increases carbide retentivity in cast iron and the first result that was in accordance with what should be expected from prior knowledge. In comparing fractures of castings from melts M-58-3 and M-74-1, it might be presumed that the amount of moisture in the air was not sufficiently concentrated to produce the desired effect in M-58-3 but about 7 grains of water per cubic foot of air was picked up by air from water at 180 degrees Fahr. so the lack of agreement in results in M-58-3 and M-74-1 admits the possible existence of an unknown having a graphitizing effect.

Since so much difficulty was experienced in obtaining concordant results when the iron was only heated to 2650 degrees Fahr. (1455 degrees Cent.), it was decided to try higher temperatures such as used in electric furnace heating of the commercial iron. The effect of temperature of heating seems to have been to produce white fracture in the M-76 series. It will be noted, however, that the fracture of the iron which was heated to 2640 degrees Fahr. (1450 degrees Cent.) and which contains 3.79 per cent total carbon plus silicon, showed a greater amount of white fracture than had been obtained in any iron up to this point with this much total carbon plus silicon. Up to this point no mention has been made of any possible effect of moisture in the atmosphere. It must be true, however, that at all times during melting the atmosphere is continually circulating throughout the charge in the furnace due to convection currents even though a cover was used on the crucible, thus bringing in contact with the large amount of surface of the hot charge a continual stream of moisture in the atmosphere. If oxides formed by bubbling steam through the melt will cause white fracture, then a continual oxidation of the surface of the charge by moist air and a reoxidation of this surface as melting proceeds should have a similar effect.

Up to this point most of the heats have been melted with very low moisture in the atmosphere. During the melting of heat M-76, however, the atmosphere contained 3.2 grains of water per cubic foot of air. The larger amount of moisture in the atmosphere might be responsible for the occurrence of the white fracture in M-76-1. On the other hand it may have been the result of the absence of the unknown graphitizing agent. Since unknown, its presence or absence

could not be discerned. It will be observed that after heating to 2850 degrees Fahr. (1565 degrees Cent.) and then cooling to 2475 degrees Fahr. (1356 degrees Cent.) before casting, the fracture becomes more mottled. This behavior is similar to that which has been described in Fig. 2 showing the effect of superheating and subsequent cooling on the production duplexed iron. The theory that non-metallic matter which might stay in solution at 2850 degrees Fahr. (1565 degrees Cent.) but which upon cooling to 2475 degrees Fahr. (1356 degrees Cent.) would precipitate out of solution and thus form nuclei for the formation of primary graphite seems to provide a satisfactory explanation for the facts in this experiment.

In most of the experiments the results obtained were not in agreement with expectations. The use of conventional measures reputed to produce white fracture did not have that effect. In a few instances the results appeared to indicate that treatment of the metal with moisture either as steam, or as water vapor in air, resulted in increased carbide retention. This was particularly true in the case of steam additions and also where the moisture was carried in with hydrogen in an experiment not described here. Whether the reducing power of hydrogen had any part in the result was subject to speculation. One way to separate reducing action from some special carbide stabilizing ability that hydrogen might have would be to use some other reducing gas—carbon monoxide for example. With this in mind, series M-80 was made in which moist carbon monoxide was bubbled through the melt. The carbon monoxide was produced by passing air through hot charcoal which produced a gas containing about 33.0 per cent CO, about 1.0 per cent CO<sub>2</sub>, and the balance nitrogen. This gas was passed through water at 180 degrees Fahr. at 12 cubic feet per hour.

In the fourth casting made from the melt treated with wet CO + N<sub>2</sub>, iron oxide was added in the furnace to augment the action of the moisture. Also in order to increase the potency of the moisture the metal was heated to 2900 degrees Fahr. (1595 degrees Cent.) before bubbling the gas through the metal. With 4 per cent total carbon plus silicon the base composition, the fracture was all gray. After passing wet CO plus nitrogen through this metal for 4 minutes at 2955 degrees Fahr. (1624 degrees Cent.), the total carbon plus silicon was 3.98 per cent and a small amount of white appeared in the fracture. After 4 more minutes of wet CO plus nitrogen, the total carbon plus silicon had been reduced to 3.73 per cent through a



0.23 per cent carbon loss. The fracture of the casting made from this iron was verging on all white. To this iron was added 0.50 per cent  $\text{Fe}_3\text{O}_4$ . This oxide appeared to dissolve immediately in the molten metal but contrary to our expectations instead of making the iron whiter it turned it completely gray. This absolute reversal of the expected result naturally gave rise to considerable speculation.



Fig. 5

M80—2.72% C, 1.28% Si, 0.38% Mn. Heated to 2900° F. 5 Gr. Water Per Cu. Ft. Air. M80-1—2.70% C, 1.28% Si, 0.40% Mn. Heated to 2955° F. Wet CO + N<sub>2</sub> Passed Through Melt—4 Min. M80-2—2.47% C, 1.26% Si, 0.42% Mn. Heated to 2900° F. Wet CO + N<sub>2</sub> Passed 4 Min. More. M80-3—2.52% C, 1.17% Si, 0.40% Mn. Heated to 2900° F. M80-2 Plus 0.5%  $\text{Fe}_3\text{O}_4$ .

Most of the experiments had gone contrary to expectations and when iron oxide caused graphitization we were beginning to think the furnace was bewitched. It has always been reported that the addition of iron oxide to molten iron increased its tendency to chill but here was a case where iron oxide acted as a graphitizer. Carbide stabilizing influence of oxygen has been encountered most frequently in connection with cast iron in thin sections and against chillers where solidification is rapid.

In order to see whether we obtained the same graphitizing result in cast iron through some obscure conditions that might be present in our particular melting procedure, we made a heat of cast iron containing 2.97 per cent carbon and 1.96 per cent silicon and made additions of 0.10 and 0.30 per cent of  $\text{Fe}_2\text{O}_3$ . Chill test castings were made without and with the oxide additions. The fractures obtained are shown in Fig. 6 and confirm the generally held conception that oxygen increases carbide retention. Because we knew that oxides either from moisture or oxide additions would increase carbide retention in cast iron when it solidified rapidly as in light sections or against chillers, we have been assuming all along that these oxides would have the same action in low carbon low silicon iron solidified slowly. We have seen that this assumption is erroneous. In spite

of a reduction in silicon in M-80-3 caused by adding iron oxide to white iron, carbide retentivity was decreased. The presence of an oxide saturated condition which would cause any further addition of oxide to exist as an emulsion of solid particles suspended in the

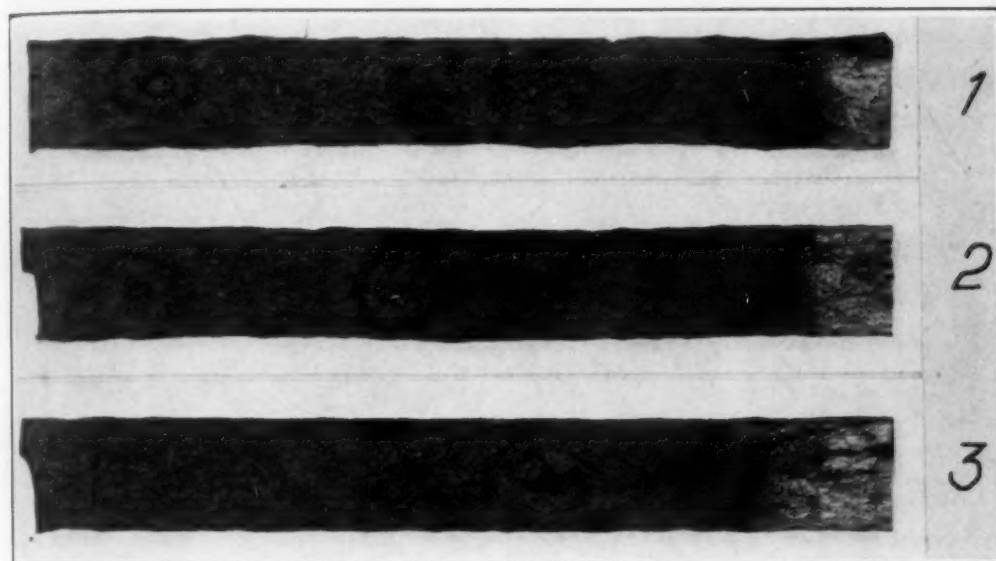


Fig. 6

- No. 1—Heated to 2740° F. 2.97% C, 0.57% Mn, 1.96% Si.  
 No. 2—Heated to 2755° F. 0.1%  $\text{Fe}_2\text{O}_3$  Added to Melt. 3.03% C, 0.57% Mn, 1.96% Si.  
 No. 3—Heated to 2770° F. 0.3%  $\text{Fe}_2\text{O}_3$  Added to Melt. 3.06% C, 0.56% Mn, 1.94% Si.

liquid iron and to act as nuclei for graphite precipitation, might be advanced as an explanation for this graphitizing action.

Upon realizing that oxide of iron acted as a graphitizer under certain conditions, we scrutinized our procedure to detect where contamination by oxide might occur. A small amount of iron sometimes solidifies on the lip of the furnace crucible and above the molten metal on the side of the crucible and oxidizes. The next metal poured would run over this oxidized metal and become contaminated. Realizing that the amount of oxide added in this way would be very small, a test was made to see what 0.1 per cent oxide added would do. Heat M-86 was devoted to this experiment. The iron was heated to 2900 degrees Fahr. (1595 degrees Cent.) and moist  $\text{CO} + \text{N}_2$  was bubbled through for 4 minutes to make it white. The room atmosphere in contact with the metal during heating and melting contained 8 grains of water per cubic foot of air. The first casting made from this heat contained 2.62 per cent carbon and 1.24 per cent silicon, or a total carbon plus silicon of 3.86 per cent, and was com-

pletely white. This result might be regarded as an indication that the atmosphere in contact with the charge while it is being heated and melted has a very important bearing on the resultant character of the metal. To this iron, which showed a white fracture in a 2-inch round, was then added 0.1 per cent  $\text{Fe}_2\text{O}_3$  and a test casting poured. This casting had a partially gray fracture as shown in M-86-1, Fig. 7. This result is similar to that obtained in heat M-80-3 where 0.50 per cent  $\text{Fe}_3\text{O}_4$  added to the melt converted a substantially white

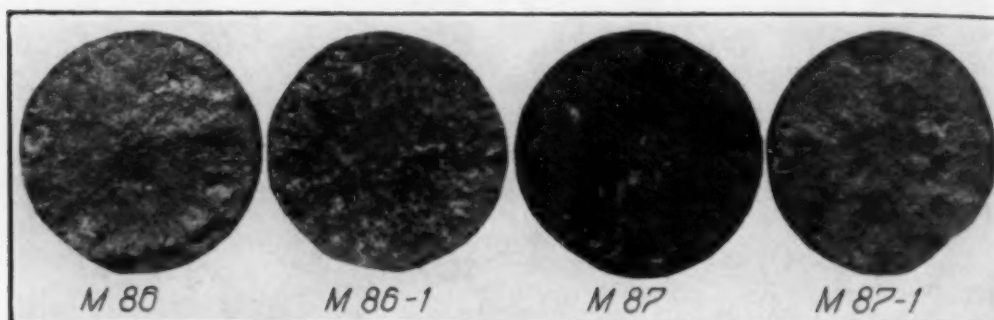


Fig. 7

M86—2.62% C, 1.24% Si, 0.39% Mn. Heated to 2900° F. 8 Gr. Water Per Cu. Ft. Air. Wet CO +  $\text{N}_2$ —4 Min. M86-1—2.69% C, 1.24% Si, 0.39% Mn. Heated to 2950° F. M86 Plus 0.1%  $\text{Fe}_2\text{O}_3$ . M87—2.68% C, 1.40% Si, 0.42% Mn. Heated to 2920° F. 4.8 Gr. Water Per Cu. Ft. Air. M87-1—2.62% C, 1.38% Si, 0.43% Mn. Heated to 2940° F. Dry Hydrogen Through Melt 4 Min.

fracture to all gray and indicates that very small amounts of oxide can upset an otherwise carefully conducted experiment. It will be noted also that the mottling consists of small diameter spots similar to those in pyramid castings from cupola metal shown in Fig. 1.

Included in Fig. 7 is another heat which was melted when the air contained 4.8 grains of water per cubic foot. With a total carbon plus silicon of 4.08 per cent, the fracture was almost completely gray but by bubbling dry hydrogen through the melt for 4 minutes at 12 cubic feet per hour, the fracture became almost white with 4.00 per cent total carbon plus silicon. This illustration was included principally for comparison with some previous heats which were treated with dry hydrogen at a lower temperature without producing any change in the fracture. This is a result similar to that obtained by Boyle who found that melting in hydrogen at a high temperature produced carbide retention although it did not at lower temperatures.

With the evidence in M-86-1 showing that a very small quantity of oxide can convert white iron to gray iron, indicating the probability that oxide formed on the sides and lip of the crucible had the power to produce a gray fracture, we looked further and found a similar



condition in the transfer ladles into which the metal was poured from the melting crucible prior to pouring the castings. Although most of the solidified metal from the previous heat was customarily removed from these ladles, there still remained more or less numerous small particles of oxidized metal imbedded in the inside surface of the crucible which were more or less porous due to repeated use. It was suspected that here was probably the condition most responsible for the frequent lack of agreement in previous tests and for the occurrence of results which were at variance with what was expected.

As a result of the discovery of the possibility of contamination by a graphitizing agent in the form of iron oxide in the transfer ladles, a practice of clay lining the transfer ladles prior to each cast was adopted. Heats M-105, M-106, M-107, and M-108 with atmospheric moisture variable were made and transferred to the molds in clay lined ladles heated for an hour at 1700 degrees Fahr. (925 degrees Cent.) before using. No disturbance occurred on pouring into these crucibles. It appears from these four heats that moisture in the atmosphere has a strong carbide retaining effect, and that the effect of moisture in contact with the charge while heating and melting is stronger than bubbling moisture through the melt—presumably because of the much greater surface area exposed by the surface of the charge than is exposed when gas bubbles travel up through only about 1½ inches of molten metal.

As a means of evaluating the influence of oxidized residue in the ladle, of the four castings made from heat M-109 two were poured after heating to 2675 degrees Fahr. (1470 degrees Cent.), one into a clay lined crucible and the other into a used unrepaired crucible. The metal for the other two castings was heated then to 2900 degrees Fahr. (1595 degrees Cent.), cooled to 2675 degrees Fahr. (1470 degrees Cent.), and poured for one casting into a clay lined crucible and for the other casting into a used unrepaired crucible. The total carbon plus silicon was too high to obtain a white fracture in the iron heated to only 2675 degrees Fahr. (1470 degrees Cent.) even though the air contained 7.6 grains of water per cubic foot but when heated to 2900 degrees Fahr. (1595 degrees Cent.) and poured into a clay lined crucible before transferring to the mold, the fracture was mostly white even with 4.04 per cent total carbon plus silicon whereas the corresponding casting which was made with metal poured into a used unrepaired crucible was completely gray with the same composition. This is rather striking evidence of the

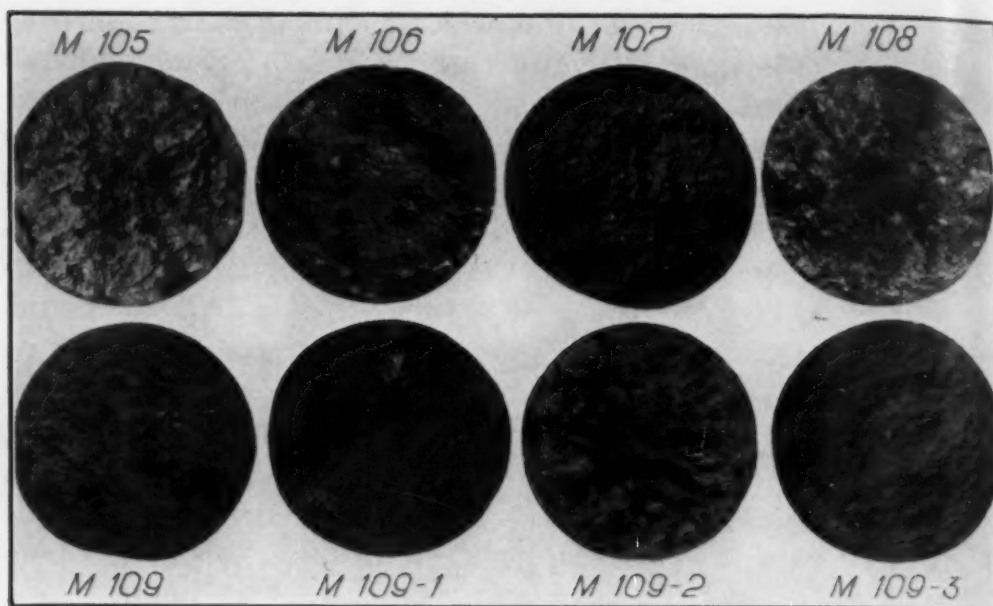


Fig. 8

M105—2.76% C, 1.21% Si, 0.38% Mn. Heated to 2900° F. Poured into Clay Lined Crucible Cast at 2675° F. 6.6 Gr. Water Per Cu. Ft. Air. M106—2.77% C, 1.31% Si, 0.38% Mn. Heated to 2900° F. Wet CO + N<sub>2</sub>—8 Min. Poured into Clay Lined Crucible Cast at 2675° F. 2.5 Gr. Water Per Cu. Ft. Air. M107—2.66% C, 1.28% Si, 0.45% Mn. Heated to 2900° F. Wet CO + N<sub>2</sub>—8 Min. Poured into Clay Lined Crucible Cast at 2675° F. 2.6 Gr. Water Per Cu. Ft. Air. M108—2.73% C, 1.31% Si, 0.45% Mn. Heated to 2940° F. Poured into Clay Lined Crucible Cast at 2650° F. 4.0 Gr. Water Per Cu. Ft. Air.

M109—2.82% C, 1.33% Si, 0.43% Mn. Heated to 2675° F. Poured into Clay Lined Crucible and Cast. 7.6 Gr. Water Per Cu. Ft. Air. M109-1—2.88% C, 1.32% Si, 0.43% Mn. Same as M109 But Poured into Used Unlined Plumbago Crucible and Cast. M109-2—2.73% C, 1.31% Si, 0.42% Mn. Heated to 2900° F. Cooled to 2675° F. Poured into Clay Lined Crucible and Cast. M109-3—2.73% C, 1.31% Si, 0.41% Mn. Same as 109-2 But Poured into Used Unlined Plumbago Crucible and Cast.

inoculating influence of solid iron oxides added to molten iron. In a later heat some of the oxidized iron pellets were collected from the transfer ladle and added to the molten iron in the furnace. This acted in the same way as the addition of oxide or pouring into an unrepaired crucible.

A number of similar tests were conducted as a means of checking this result and in M-111 and M-112 series the metal poured into the unrepaired plumbago crucible showed an appreciable increase in the amount of gray in the fracture. It will be noted also that the mottles are small in size, a characteristic of iron which has been graphitized by adding iron oxide. The white fractures of the castings poured from clay lined crucibles were in striking contrast to the gray fractures obtained with previous heats of similar composition or of lower total carbon plus silicon made before the discovery of the inoculating action of iron oxide. In only one case (M-110 series) where the metal was melted on a day when the air contained

8 grains of water per cubic foot did pouring into an unrepaired crucible fail to cause gray fracture. This was presumed to be for the reason that the influence of the high moisture in the air was so strong in producing carbide stability that there was not enough oxide in the crucible to cause graphitization. Attention is called especially to heat M-110 because with 4.02 per cent total carbon plus silicon

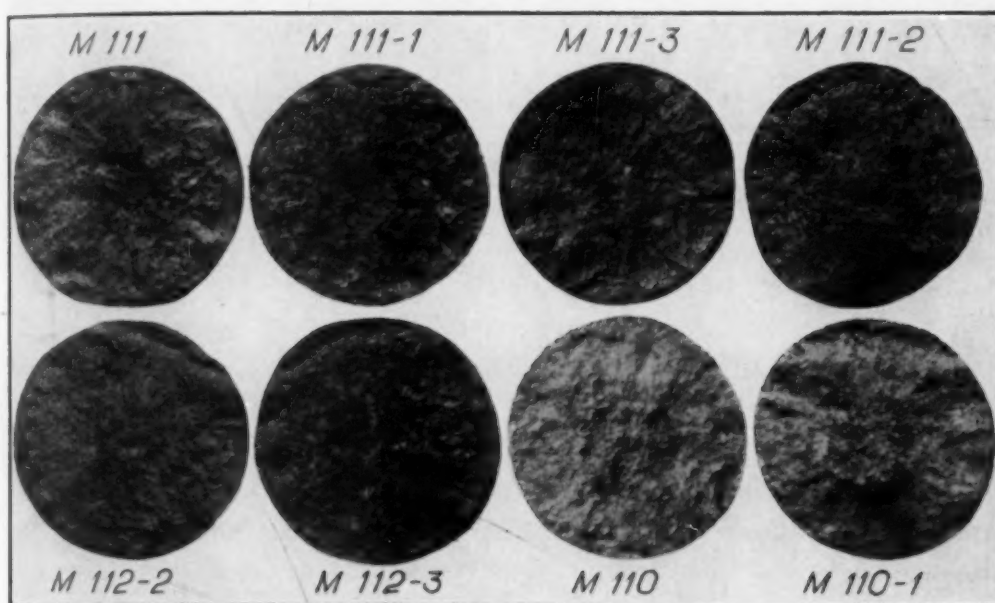


Fig. 9

M111—2.55% C, 1.16% Si, 0.39% Mn. Heated to 2680° F. Poured into Clay Lined Crucible and Cast. 5.6 Gr. Water Per Cu. Ft. Air. M111-1—2.51% C, 1.11% Si, 0.39% Mn. Heated to 2680° F. Poured into Used Unlined Plumbago Crucible and Cast. M111-3—2.53% C, 1.11% Si, 0.34% Mn. Heated to 2900° F. Cooled to 2680° F. Poured into Clay Lined Crucible and Cast. M111-2—2.41% C, 1.06% Si, 0.34% Mn. Heated to 2900° F. Cooled to 2680° F. Poured into Used Unlined Plumbago Crucible and Cast.

M112-2—2.58% C, 1.08% Si, 0.34% Mn. Heated to 2890° F. Cooled to 2650° F. Poured into Clay Lined Crucible and Cast. 3.8 Gr. Water Per Cu. Ft. Air. M112-3—2.58% C, 1.08% Si, 0.34% Mn. Heated to 2890° F. Cooled to 2650° F. Poured into Used Unlined Plumbago Crucible and Cast. M110—2.83% C, 1.19% Si, 0.39% Mn. Heated to 2645° F. Poured into Clay Lined Crucible and Cast. 8.0 Gr. Water Per Cu. Ft. Air. M110-1—2.63% C, 1.16% Si, 0.39% Mn. Heated to 2645° F. Poured into Unlined Used Plumbago Crucible and Cast.

and only heated to 2645 degrees Fahr. (1450 degrees Cent.), the fracture was clear white which is equivalent to the carbide retaining power of metal melted for production by the duplex process. From this point on all experiments were conducted with freshly clay lined crucibles preheated to 1700 degrees Fahr. (925 degrees Cent.) in an electric furnace in order to prevent inoculation by the oxide particles remaining in the crucible from the previous melt.

Because some of our results had pointed to the fact that the nature of the atmosphere surrounding the charge while heating up



and melting was more important than the passage of various atmospheres through the metal in small quantities or the nature of the atmosphere in contact with the surface of the molten metal after all the charge had been melted, a melting procedure was adopted whereby the desired type of atmosphere was supplied in a continuous stream into the crucible at a sufficient rate so that none of the extraneous atmosphere could intrude. The gas was introduced through a quartz tube penetrating the crucible cover. With this type of control whereby any inoculating effect of extraneous oxides was prevented, it was possible to obtain more concordant results. These results indicated that moisture and hydrogen have a positive power to overcome the graphitizing influence of increased carbon and silicon. The prevention of contamination of the melt by solid iron oxide is even more important to the production of white fracture with high silicon and carbon than is the presence of moisture.

The powerful carbide retaining effect of moisture when the graphitizing influence of solid iron oxides is excluded are shown in Fig. 10, heats M-120 and M-121. In heat M-120 steam was introduced into the crucible during heating and melting and the iron was heated to only 2675 degrees Fahr. (1470 degrees Cent.) and with a total carbon plus silicon of 3.85 per cent the fracture was entirely white. In heat M-121 moist air at 12 cubic feet per hour was introduced into the crucible. This air contained 7.2 grams of moisture per cubic foot of air as a result of passing through water at 180 degrees Fahr. In this casting, which contained 3.93 per cent total carbon plus silicon, the fracture was completely white when the metal had been heated to only 2675 degrees Fahr. (1470 degrees Cent.). This is certainly a remarkable difference from results in heat M-37-2 where with a total carbon plus silicon of 3.42 per cent and heated to 2680 degrees Fahr. (1470 degrees Cent.) the fracture was completely gray. The difference in carbide retentivity is as great as that represented by the difference in behavior between air furnace malleable iron and duplexed cupola electric furnace malleable iron. It is not contended that the carbide retentivity in cupola malleable iron is the result of large quantities of moisture in the atmosphere. A pronounced difference between crucible melting and cupola melting may be found in the degree of exposure of the molten metal and the charge, to gaseous elements. In the cupola there is a constant rush of gases at high velocity coming in contact with the charge being heated and with the molten metal broken up into small drops present-

ing a tremendous surface area for reaction with these gases. It is believed that the concentration of moisture in the gas is not as important as how much moisture is brought into contact with each unit weight of molten metal. The cupola gases need contain only a small

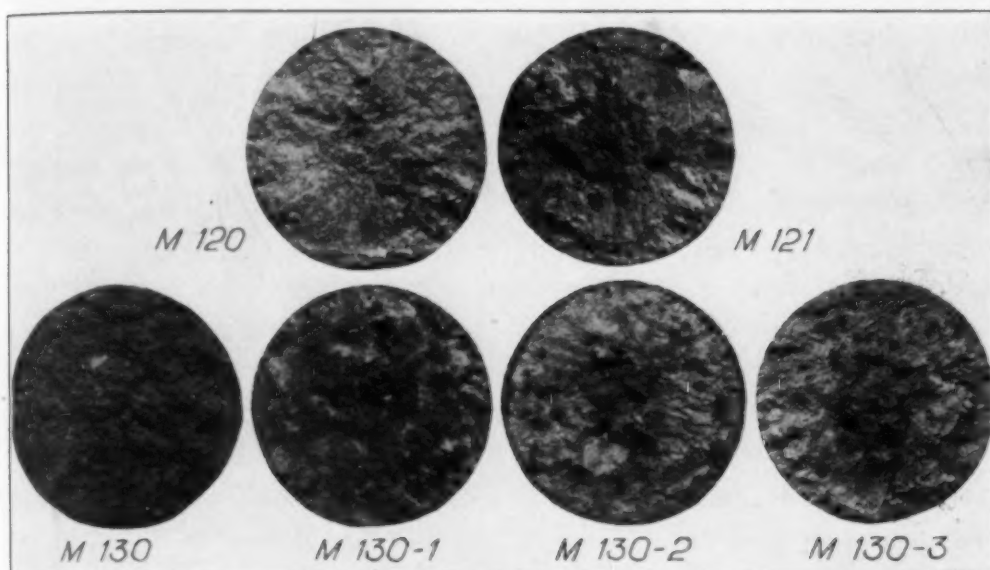


Fig. 10

M120—2.50% C, 1.35% Si, 0.40% Mn. Steam Maintained in Crucible During Heating and Melting. Heated to 2675° F. M121—2.62% C, 1.31% Si, 0.43% Mn. Wet Air at 12 C.F. Per Hour into Crucible During Heating and Melting. 7.2 Gr. H<sub>2</sub>O Per C.F. Air Heated to 2675° F.

M130—2.70% C, 1.26% Si, 0.41% Mn. Dry Air Through Act. Al<sub>2</sub>O<sub>3</sub> into Crucible at 6 C.F. Per Hour During Heating and Melting. Heated to 2760° F. M130-1—2.70% C, 1.26% Si, 0.40% Mn. Heated to 2860° F. Poured at 2700° F. M130-2—2.70% C, 1.26% Si, 0.40% Mn. Heated to 2910° F. Poured Immediately. M130-3—2.39% C, 1.26% Si, 0.40% Mn. Metal in Ladle Left from M130-2. Cooled to 2700° F. and Poured.

amount of moisture in order to have each unit weight of molten iron come in contact with just as much water as could be obtained in a crucible melt by having a gas highly saturated with moisture in contact with the charge.

In Fig. 1 it will be observed that the pyramid castings made from cupola metal, which had not been superheated in the electric furnace, showed a greater mottling tendency and small diameter mottles characteristic of a metal to which iron oxide had been added. It is believed that this is not an indication of anything that happened in the cupola. The metal for casting these pyramid castings was procured by pouring from a 5-ton transfer ladle into a hand shank ladle. It is entirely possible and probable that this hand shank, having been used for making other castings, contained some oxidized metal in the bottom which was capable of inoculating the metal sufficiently

to be responsible for the increased amount of mottling. On the other hand, the molds for pyramid castings made from electric furnace iron were placed on the production conveyor line and received metal from the regular pouring ladles, which are kept replenished with hot metal frequently enough so that they always contain molten metal, so there is no opportunity for contamination with solid iron oxide. Fig. 10 also shows, for comparison with M-121, fractures of castings made from iron melted in an atmosphere of dry air. In M-130 the crucible was kept supplied with dry air at 6 cubic feet per hour during heating and melting. The moisture was removed by passing air through activated alumina. With a total carbon plus silicon of 3.96 per cent, the fracture was gray even when heated to 2760 degrees Fahr. (1515 degrees Cent.) and, even when heated to 2860 degrees Fahr. (1570 degrees Cent.), the fracture contained only about 25 per cent white. By heating to 2900 degrees Fahr. (1595 degrees Cent.) and pouring immediately, the fracture was practically all white and when allowed to cool to 2700 degrees Fahr. (1480 degrees Cent.) and cast it was still all white and did not show the ring of gray which characterized the iron cast from 2900 degrees Fahr. (1595 degrees Cent.). Fractures of series M-130 show that even with dry air white fracture in a 2-inch round casting may be obtained by heating to 2900 degrees Fahr. (1595 degrees Cent.) with a composition as high as 3.96 per cent total carbon plus silicon if contaminating solid oxide influence is prevented. The chief culprit responsible for gray fracture with low carbon plus silicon appears to be solid iron oxide. That this is part of the reason for the low carbon and silicon maintained in air furnace melting, appears plausible when we remember the universal custom of emptying the air furnace with hand shank ladles while the stream runs continuously out of the furnace, necessitating a long line of pourers waiting their turn to catch a ladleful and, while waiting, the residue clinging to the ladle lining oxidizes and prepares to do a good job of inoculating a perfectly good white iron coming from the furnace.

Two series of castings are shown in Fig. 11 which were made to show the effect of using carbon dioxide as the furnace atmosphere in place of air. In series M-129 the carbon dioxide was dried by passing through activated alumina, and in series M-128 it contained 6.6 grains of water per cubic foot by passing through water at 80 degrees Fahr. Both of these gases were passed into the crucible at 6 cubic feet per hour. Although this provides a rather small amount

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of moisture per pound of metal melted, the fractured castings indicate that there is an appreciable effect of this moisture in producing a whiter fracture in the M-128 series. M-128 is comparable with

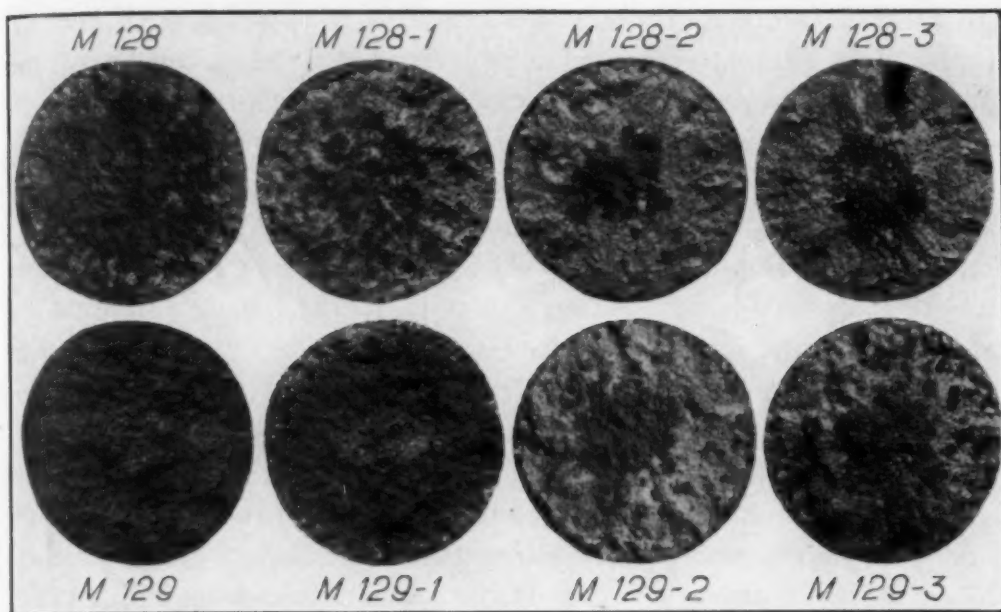


Fig. 11

M128—2.64% C, 1.26% Si, 0.43% Mn. Heated to 2730° F.	M128-1—2.62% C,
1.30% Si, 0.43% Mn. Heated to 2800° F. Poured at 2700° F.	M128-2—2.46% C,
1.30% Si, 0.44% Mn. Heated to 2900° F. Poured Immediately.	M128-3—2.50% C,
1.33% Si, 0.43% Mn. Heated to 2900° F. Poured at 2700° F.	
100% CO <sub>2</sub> With 6.6 Gr. H <sub>2</sub> O Per Cu. Ft. into Crucible at 6 Cu. Ft. Per Hour While Heating and Melting.	
M129—2.55% C, 1.33% Si, 0.43% Mn. Heated to 2650° F.	M129-1—2.52% C,
1.30% Si, 0.41% Mn. Heated to 2750° F. Poured at 2700° F.	M129-2—2.48% C,
1.35% Si, 0.42% Mn. Heated to 2910° F. Poured Immediately.	M129-3—2.53% C,
1.35% Si, 0.41% Mn. Heated to 2900° F. Poured at 2520° F.	
100% CO <sub>2</sub> —Dry (Through Activated Al <sub>2</sub> O <sub>3</sub> ) into Crucible at 6 Cu. Ft. Per Hour While Heating and Melting.	

M-129-1. M-129-1 has somewhat less total carbon plus silicon than M-128, and was also heated to a slightly higher temperature but, in spite of both of these things which would increase carbide retention, the fracture of M-129-1 contains less carbide than M-128.

An interesting comparison may be made between series M-129 and M-130 which indicates that dry carbon dioxide has no greater carbide retaining influence than does dry air. It had been presumed that the carbon dioxide would give up an oxygen atom to more readily oxidize the iron than would the dry air but the results do not support this view. On the other hand the carbide retaining power produced by 6.6 grains of water per cubic foot of CO<sub>2</sub> in 6 cubic feet of CO<sub>2</sub> per hour is considerably less than in M-121 when air passed

through water at 180 degrees Fahr. enters the crucible at 12 cubic feet per hour.

As a result of these tests we have collected enough evidence to make us appreciate the wisdom from now on of regarding more carefully the production of hard iron for making malleable iron from the standpoint of the influence of water vapor and that of contamination of solid iron oxide. When we attempt to explain the mechanism of these two influences we are confronted with the thought that iron oxide is probably the active ingredient in both cases. In one form it causes carbide stability and in another it causes graphitization. A difference that might be pointed to is that the oxide formed as a result of moisture decomposition probably forms a different kind of oxide than that formed by air when molten iron is solidifying and cooling from the freezing temperature.

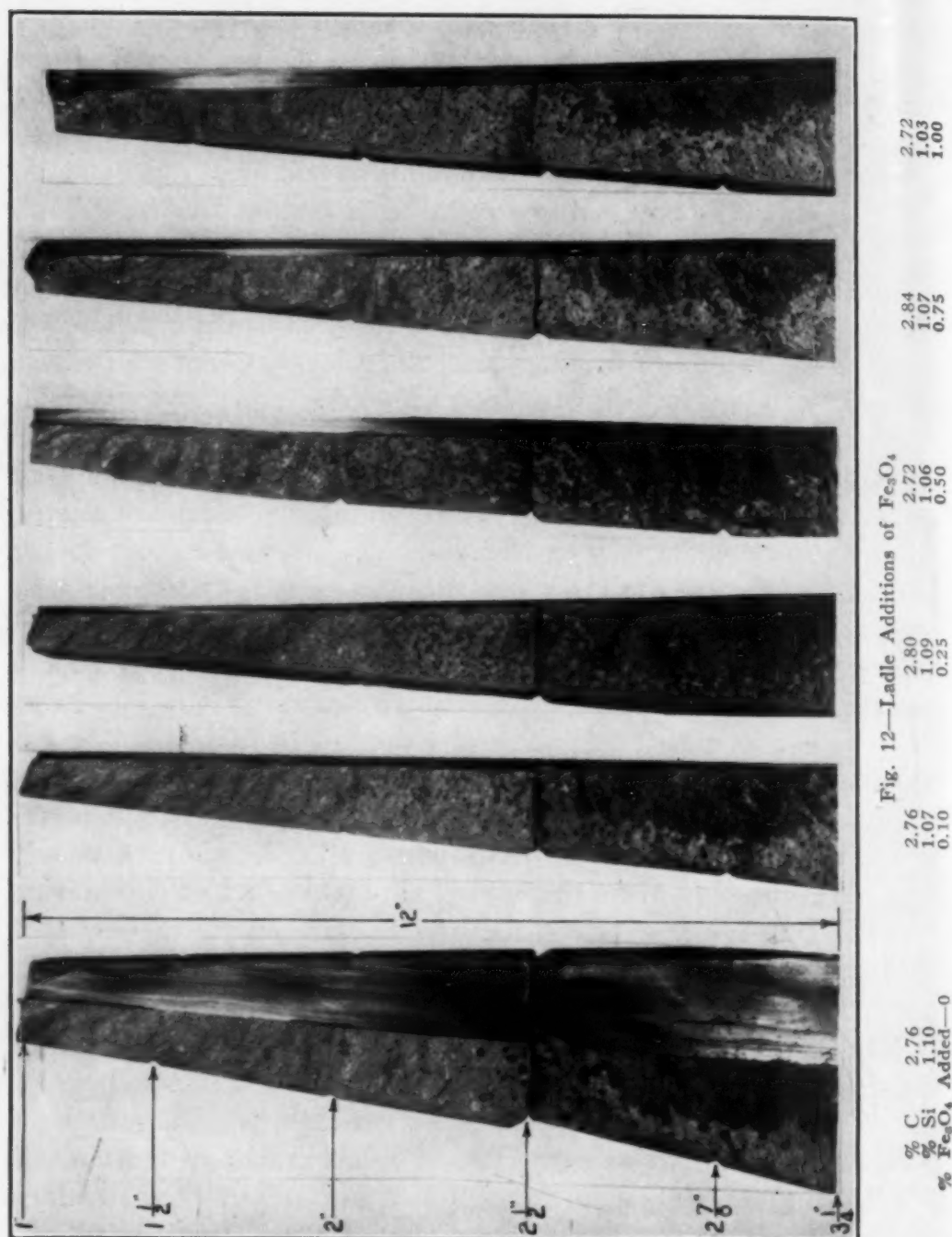
It should be pointed out also that nascent hydrogen formed when water decomposes may play some part in the stabilizing effect. The problem is no doubt very complicated and probably has to do with whether oxides or silicate slags are dissolved or whether they are out of solution and if they are out of solution whether they are molten or solid. It may also involve differences in solubility or reaction of the different forms of iron oxide. This part of the problem must be left to someone who will take these phases one at a time and set up carefully controlled experiments to get satisfactory answers. For the present we are content to know what practical results may be obtained. The findings of this work apply not only to the production of white iron for conversion to malleable iron but also to the production of high strength cast iron. The same influences must be taken into consideration in induction furnace melting of gray iron. It appears that experimental melting in high frequency furnaces will yield more useful results if careful control of the atmosphere surrounding the furnace charge and of the condition of casting ladles is maintained.

The importance of being able to obtain white iron with a high carbon plus silicon content obviously resides in the shorter time required for annealing. The time required to convert white iron to malleable iron by annealing has been progressively reduced during the past fifteen years as a result of a study of the time to reach equilibrium at various stages of the annealing process; by reducing the mass of pots and castings closely stacked together through which heat had to penetrate; and in some instances by increasing the silicon

content at the expense of a reduction in carbon content. The lighter the casting and the faster the metal solidifies, the higher may be the silicon content without formation of primary graphite in the casting and consequently the faster will the annealing be completed. Following this relationship still further, we find that still more rapid solidification such as that obtained in a metal mold permits the use of such high silicon content that it becomes only necessary to heat the casting up to 1700 degrees Fahr. (925 degrees Cent.) and allow it to cool to obtain complete annealing. We are ordinarily concerned however with the production of castings in sand molds, castings which have sections up to 1 inch thick which cool or solidify at a rate comparable with that of a 2-inch round bar. For this purpose, iron melted in the cupola with a charge of steel scrap, foundry returns, and silvery pig iron, and heated to pouring temperature in a direct arc electric furnace, may contain 2.70 to 2.75 per cent carbon, 1.15 to 1.20 per cent silicon, 0.38 to 0.42 per cent manganese, about 0.05 per cent phosphorus and 0.09 to 0.11 per cent sulphur, and will usually produce a white fracture in a 2-inch round casting. We note over a period of time that within the composition range just mentioned that the response to annealing treatment varies more than could be explained by variation in the amount of carbon, silicon, manganese, sulphur, and phosphorus content. The causes for variation in response to annealing treatment may possibly be found to be closely related or identical with the causes of carbide retentivity during solidification. Since we have discussed at some length the effect of adding iron oxide to molten iron, upon solidification structure in laboratory melts, the picture should be completed with its effect on production iron, both as to solidification and as to response to annealing treatment.

A number of pyramid castings were poured from regular production duplexed iron to which had been added various amounts of  $\text{Fe}_3\text{O}_4$ . Fig. 12 shows the longitudinal fracture of these castings compared with the fracture of a casting made from the regular production iron. The casting which was 3.25-inch square at the large end had to be cut with a rubber wheel half way through before it could be fractured so that the photograph shows only one-half of the cross section of the casting. The effect of the oxide addition here is essentially the same as that which was produced in laboratory melts in the high frequency induction furnace. The areas containing primary graphite which constitute the mottling are very much smal-

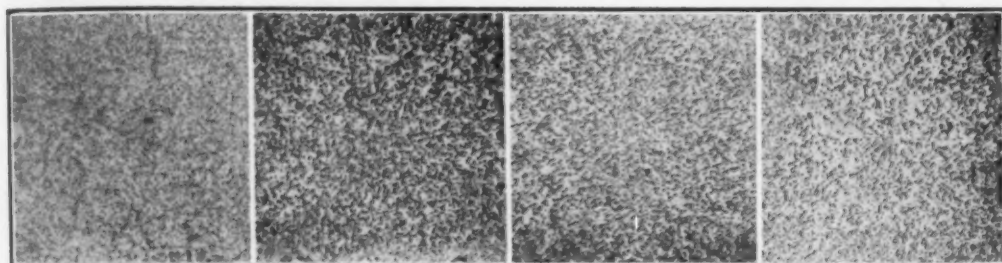




ler in diameter than in the regular iron and the mottling extends into smaller sections when up to 0.50 per cent  $\text{Fe}_3\text{O}_4$  is added. Above that amount the mottling tendency is very little greater than in the standard iron. This apparent reversal of influence should receive additional investigation. This confirms the evidence obtained from laboratory melts previously described showing that iron oxide added

to the ladle acts as a graphitizer and not as a carbide stabilizer as has been commonly supposed.

The influence of these oxide additions on response to annealing treatments is portrayed in Fig. 13 showing the entire cross section of 7/8-inch square bars after annealing. These photographs show the distribution of the temper carbon spots. Several investigators in recent years have discussed how the rate of carbide elimination dur-



During Annealing—Heated at 36° F. Per Hour—1520° to 1700° F.

HH	JJ	LL	MM
2.76% C	2.80% C	2.84% C	2.72% C
1.10% Si	1.09% Si	1.07% Si	1.03% Si
0.37% Mn	0.36% Mn	0.36% Mn	0.34% Mn
0.11% S			
0.05% P			
No Fe <sub>3</sub> O <sub>4</sub> Added	0.25% Fe <sub>3</sub> O <sub>4</sub> Added	0.75% Fe <sub>3</sub> O <sub>4</sub> Added	1.00 Fe <sub>3</sub> O <sub>4</sub> Added

During Annealing—Heated 400° F. Per Hour—1300° to 1700° F.

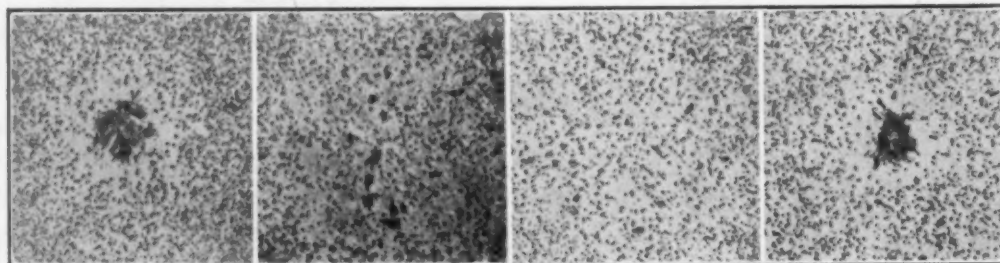


Fig. 13

ing annealing decreases as the distance between temper carbon spots increases. The four pictures at the top show the effect of iron oxide additions upon the formation of temper carbon when heating to 1700 degrees Fahr. (925 degrees Cent.) in the annealing cycle is at 36 degrees per hour from 1520 to 1700 degrees Fahr. (825 to 925 degrees Cent.), which has been adopted as the standard method of heating because it produces fine close packed temper carbon spots and consequently facilitates rapid annealing.

It will be observed that even with this slow heating the iron oxide additions tend to make the temper carbon spots larger and

further apart, especially toward the center of the casting where the solidification rate is slower. It is presumed that in still larger sections that this tendency will be more pronounced. When the same castings are heated at 400 degrees per hour to 1700 degrees Fahr. (925 degrees Cent.) in the annealing cycle, the iron oxide has a similar influence and makes the temper carbon distribution even less desirable than the regular iron shows after this fast heating.

The microstructure in these  $\frac{7}{8}$ -inch square bars associated with annealing of iron to which iron oxide has been added is shown in Fig. 14. Photomicrographs 1, 2 and 3 show the effect of the iron oxide addition upon the speed of carbide elimination when heated rapidly to 1700 degrees Fahr. (925 degrees Cent.). This is apparently a function principally of the size and distance between temper carbon spots which appears to increase directly as the amount of iron oxide added. Photomicrographs 4, 5 and 6 show the effect of the iron oxide upon the structure after the castings have been subjected to the standard annealing cycle which includes heating at 36 degrees per hour from 1500 to 1700 degrees Fahr. (815 to 925 degrees Cent.), holding for 8 hours at 1700 degrees Fahr. (925 degrees Cent.), cooling to 1400 in two hours and cooling from 1400 to 1300 degrees Fahr. (760 to 705 degrees Cent.) at 7 degrees per hour. Even with this slow heating it will be observed that the temper carbon spots are greatly enlarged by the iron oxide addition. Although this apparently has not retarded decomposition of massive cementite enough to prevent complete elimination in 8 hours at 1700 degrees Fahr. (925 degrees Cent.), it has however retarded second stage graphitization to the extent that considerable pearlite remained after cooling at 7 degrees per hour. We see, therefore, that the action of iron oxide additions is to increase the tendency to mottle and at the same time to make it more difficult to anneal, which is contrary to the characteristics that would result if increased mottling tendency were produced by the addition of some graphitizing agent such as silicon, copper, or the like. With the addition of such elements, the mottling tendency would increase so that the iron would be only useful in lighter sections but because of the addition of these graphitizing agents the rate of carbide elimination in annealing would be speeded up. We see, therefore, that addition agents cannot be referred to as carbide stabilizing agents without specifying whether they stabilize carbides during solidification or during annealing, or both. In the case of iron oxide, we have the paradoxical situation of a material that has a net



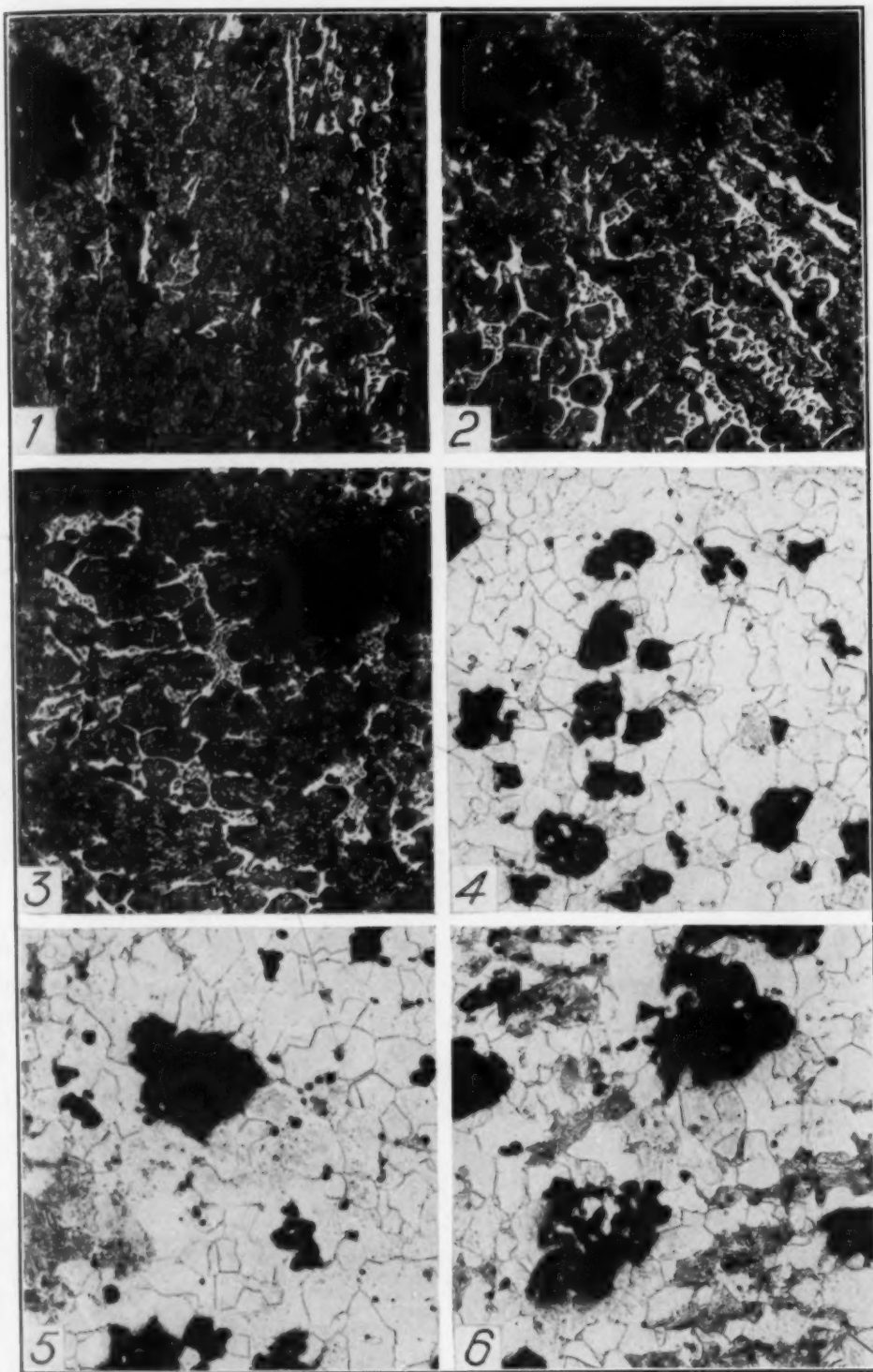


Fig. 14—Annealed With Standard Cycle—Including Heating at 36° F. Per Hour—1500° to 1700° F.

No. 1—Standard Iron HH. 2.76% C, 1.10% Si. HH Plus 0.10%  $\text{Fe}_3\text{O}_4$ .  
 No. 2—HH Plus 0.25%  $\text{Fe}_3\text{O}_4$  Ladle Addition. 2.80% C, 1.09% Si. HH Plus 0.25%  $\text{Fe}_3\text{O}_4$ .  
 No. 3—HH Plus 1.00%  $\text{Fe}_3\text{O}_4$  Ladle Addition. 2.72% C, 1.03% Si. HH Plus 0.75%  $\text{Fe}_3\text{O}_4$ . Heated to 1700° F. at 400° F. Per Hour, Held 8 Hours and Air Cooled. No. 4—HH Plus 0.10%  $\text{Fe}_3\text{O}_4$ . No. 5—HH Plus 0.25%  $\text{Fe}_3\text{O}_4$ . No. 6—HH Plus 0.75%  $\text{Fe}_3\text{O}_4$ .

influence like a graphitizing agent during solidification but which acts like a carbide stabilizer during annealing.

The explanation that occurs to us for the increased mottling caused by iron oxide additions is suggested by Von Keil's silicate slime theory and that is that the iron oxide combines with silicon and manganese forming iron manganese silicates which act as nuclei for graphite precipitation during solidification. The mechanism by which carbide elimination is prevented during annealing is thought to be due principally to the increased distance between temper carbon spots. As a hypothesis to explain why iron oxide causes fewer temper carbon spots to form, we might presume that iron oxide in solution interferes with the ready diffusion of carbon in solution so that precipitation occurs from a few regions of segregated high carbon instead of from many places as it should if solution of carbon were uniform throughout. We might further speculate that because oxide dissolved in iron reduces the solubility of iron carbide in iron, it thus reduces the diffusibility of carbon from carbide areas to temper carbon areas. In second stage graphitization the presence of oxide in solution may increase the tendency for carbide to precipitate from solution, thus retarding annealing because graphitization proceeds more rapidly from austenite than directly from carbide out of solution. Later some experiments with copper oxide additions may throw some light on whether oxides would retard annealing if the distance between temper carbon spots were not increased.

Presumably the action of the oxygen introduced into iron depends entirely upon the form in which it is when introduced into the iron. For example, oxygen introduced as  $\text{SiO}_2$  or  $\text{Al}_2\text{O}_3$  might conceivably combine with silicates already present in the molten iron to affect the freezing characteristics by forming nonmetallic nuclei but such oxide would not have any influence upon annealability because of its inertness when tightly bound to the silicon or aluminum.

Oxygen added in combination with any metal that has a weaker bond for oxygen than iron, or silicon, will combine with the iron or silicon but the metal freed from the oxygen will have its individual effect upon the freezing and annealing process and thus may be strong enough to counteract the effect of oxygen. As an example we might consider the effect of oxygen added as copper oxide.

Before discussing the effect of copper oxide it would be desirable to provide some background regarding the influence of copper by itself without oxygen. The effect of copper by itself has been re-

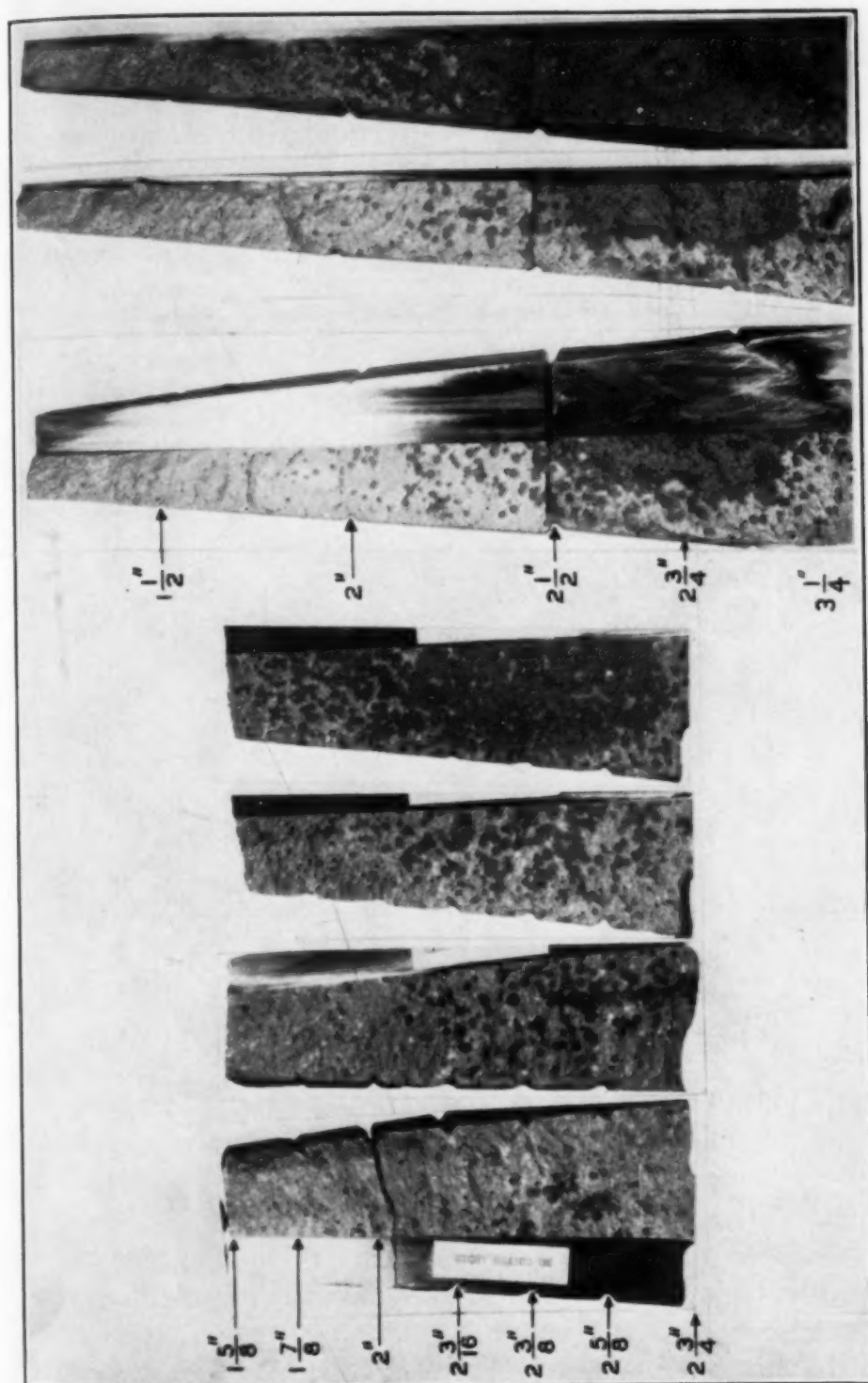


Fig. 15

No Copper  
2.77% C, 1.05% Si, 0.34% Mn, 0.11% S, 0.05% P  
Made With Pig Iron A—Large Temper Carbon

No Copper  
2.73% C, 1.16% Si, 0.38% Mn, 0.105% P, 0.048% S  
Made With Pig Iron B—Small Temper Carbon

ported a number of times. Smith and Palmer<sup>3</sup> and Lorig and Smith<sup>4</sup>

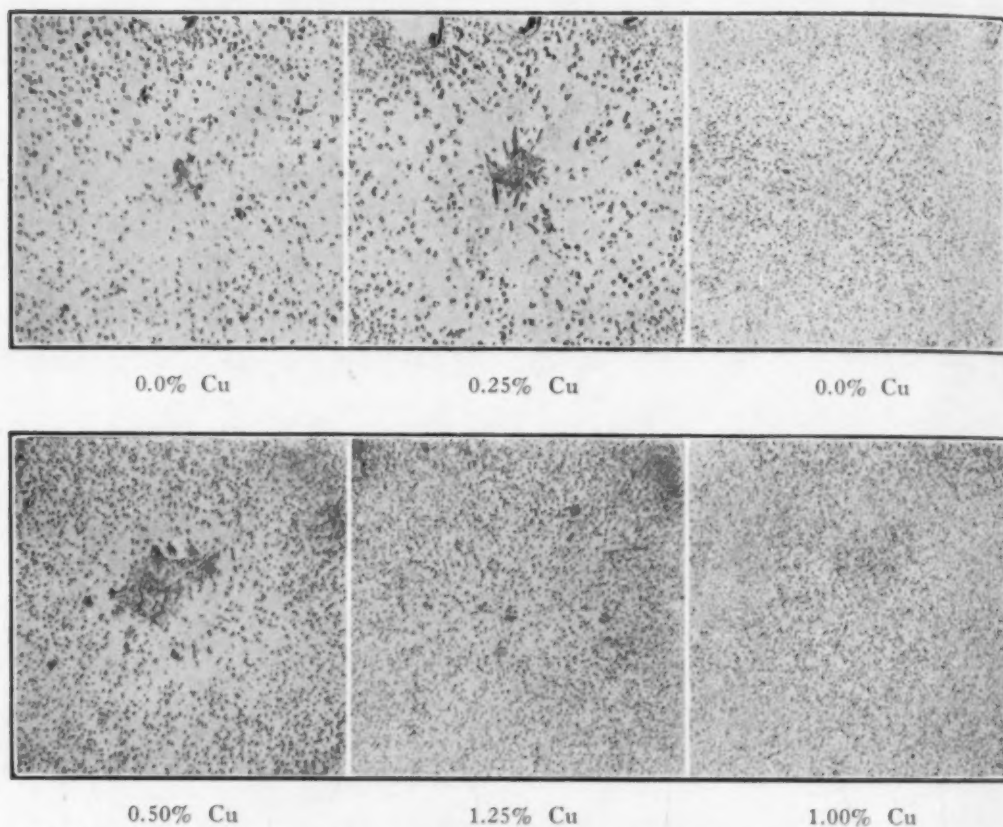
<sup>3</sup>Cyril S. Smith and Earl W. Palmer, American Institute of Mining and Metallurgical Engineers, Technical Publication 603, Metals Technology, February, 1935.

<sup>4</sup>C. H. Lorig and C. A. Smith, *Foundry Trade Journal*, January 31, 1935, p. 95.



have told how copper speeds up graphitization and Smith and Palmer mentioned the refining effect upon temper carbon spot size.

They did not show the effect of copper on tendency to mottle nor the progressive effect on temper carbon distribution with increas-



Made With Pig Iron A—2.77% C, 1.05% Si, 0.34% Mn.

Made With Pig Iron B—  
2.73% C, 1.16% Si, 0.38% Mn.

Fig. 16—Effect of Heating at 400° F. Per Hour—From 1300° to 1700° F. in Anneal.

ing copper content. Figs. 15, 16 and 17 show the effect on mottling tendency and temper carbon distribution produced by copper additions to iron made three months apart using different pig irons in the charge. Although the pyramid castings made from Pig Iron A were smaller than those made from Pig Iron B and the silicon was eleven points higher in the latter, the increase in mottling produced by the copper was about the same in both cases when compared at the 2-inch section. 0.50 per cent copper added produced a slight increase in mottling in the A iron while 0.50 per cent copper produced practically no increase in the B iron. 1.00 per cent copper produced about the same amount of mottling in both cases when compared at the 2-inch sections in both castings. Comparing the

mottling in these pyramid castings with that in castings in Fig. 1 wherein the most mottled casting represented iron which was satisfactory for castings, it is obvious that if it were desired for some reason to use copper in malleable iron, up to 1.25 per cent could be used without incurring too much mottling for most of the castings made today for automotive consumption.

The interesting phenomena observed in a comparison of these two heats is the difference in temper carbon distribution occurring after rapid heating to 1700 degrees Fahr. (925 degrees Cent.) in the annealing cycle. In iron made with Pig Iron A the temper carbon

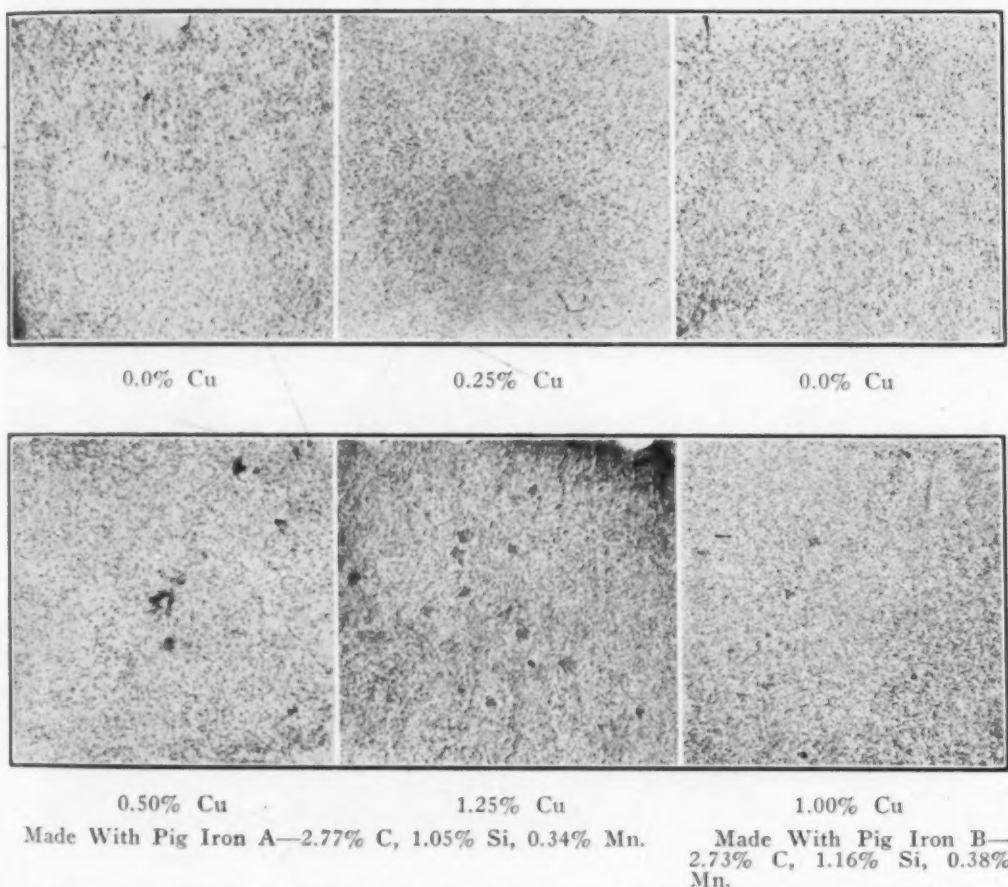


Fig. 17—Effect of Heating at 36° F. Per Hour—1520° to 1700° F. in Anneal.

is coarse and sparse in contrast to the fine densely packed temper carbon in the samples cast from iron made with Pig Iron B.

With each increase in the amount of copper added to the A iron, the temper carbon in the samples heated rapidly to 1700 degrees Fahr. (925 degrees Cent.) showed a refinement of temper carbon size beginning at corners and edges where the solidification rate was

fastest. This effect gradually penetrated toward the center till with 1.25 per cent copper, the temper carbon was almost as fine throughout as the samples heated slowly to 1700 degrees Fahr. (925 degrees Cent.).

The A iron is the same type as the iron discussed by the author earlier this year<sup>5</sup> in respect to effect of heating rate in annealing cycle upon size and distribution of temper carbon spots. Slow heat-

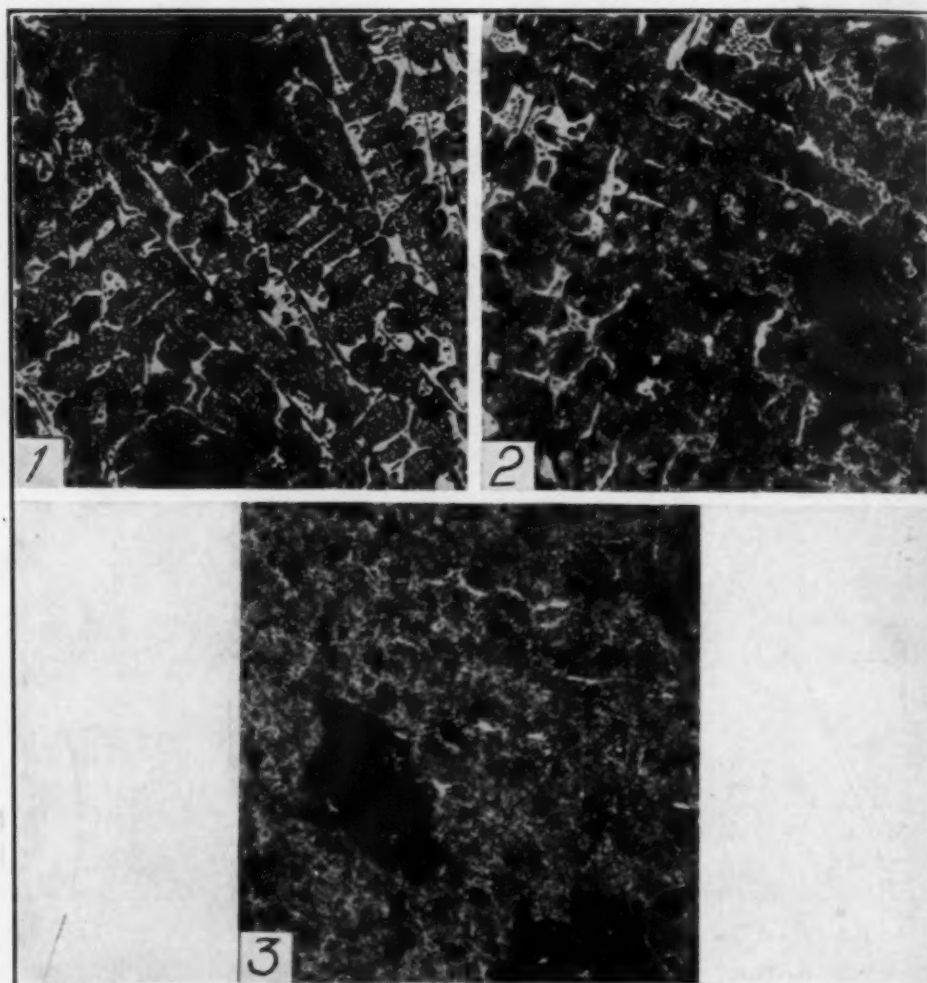


Fig. 18—Heated to 1700° F. at 400° F. Per Hour, Held 8 Hours, Then Air Cooled.

- 1—Standard Iron H. 2.77% C, 1.05% Si.
- 2—H Plus 0.50% Copper.
- 3—H Plus 1.00% Copper.

ing was found to produce many small temper carbon spots close together but fast heating caused large widely separated carbon spots. The small temper carbon is preferred because with this type of

<sup>5</sup>A. L. Boegehold, "Factors Influencing Annealing Malleable Iron," American Foundrymen's Association, Preprint 38-24, May, 1938.



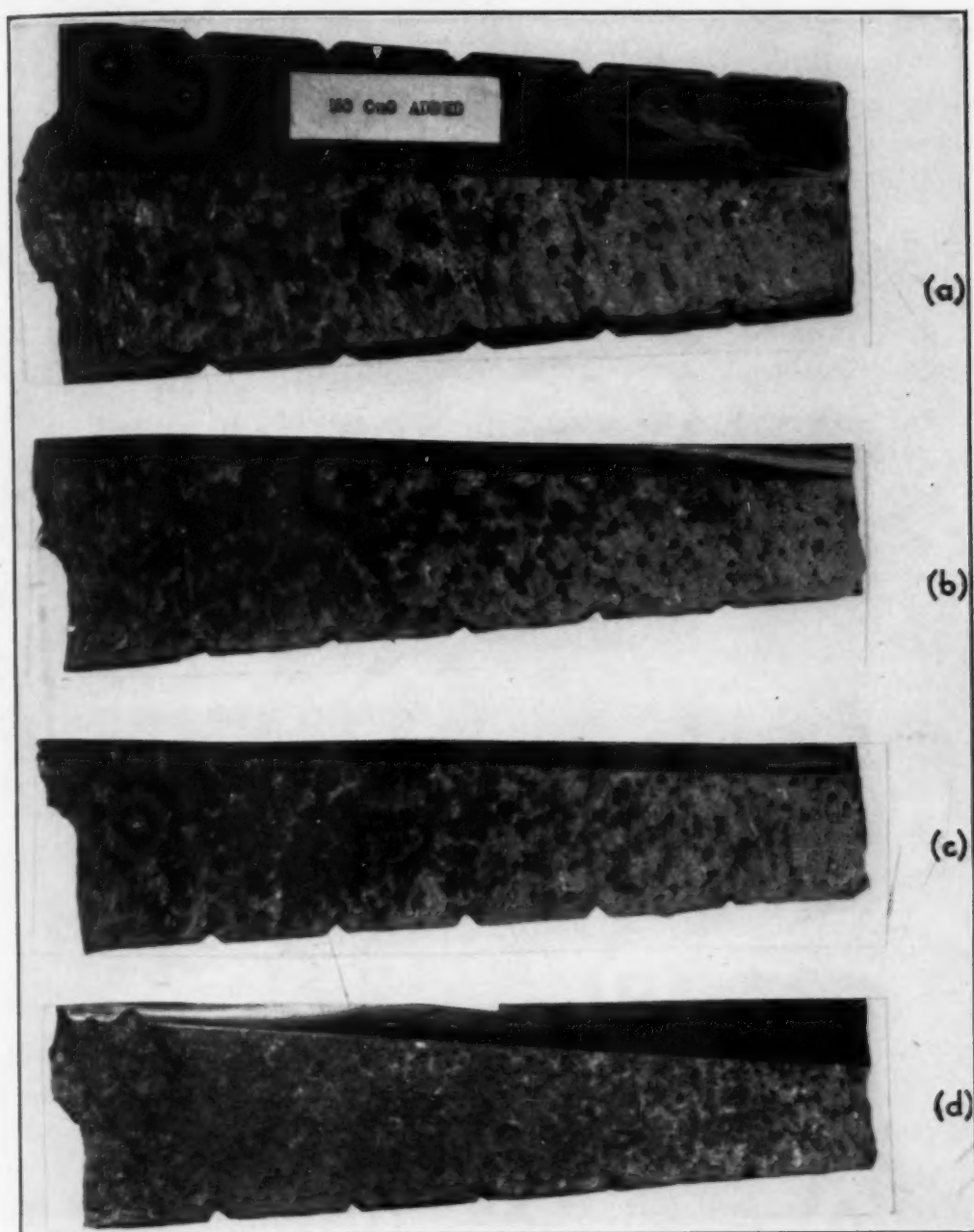


Fig. 19

- |                     |                     |
|---------------------|---------------------|
| (a) Standard Iron   | (b) 0.25% CuO Added |
| (c) 0.50% CuO Added | (d) 1.00% CuO Added |
| 2.75% C             | 1.16% Si            |
|                     | 0.37% Mn            |

structure carbide elimination is much more rapid. The B iron is a type not previously reported in the literature. It develops small temper carbon spots even when heated rapidly to 1700 degrees Fahr. (925 degrees Cent.) and since it is not sensitive to rate of heating to 1700 degrees Fahr. (925 degrees Cent.) in the annealing operation,

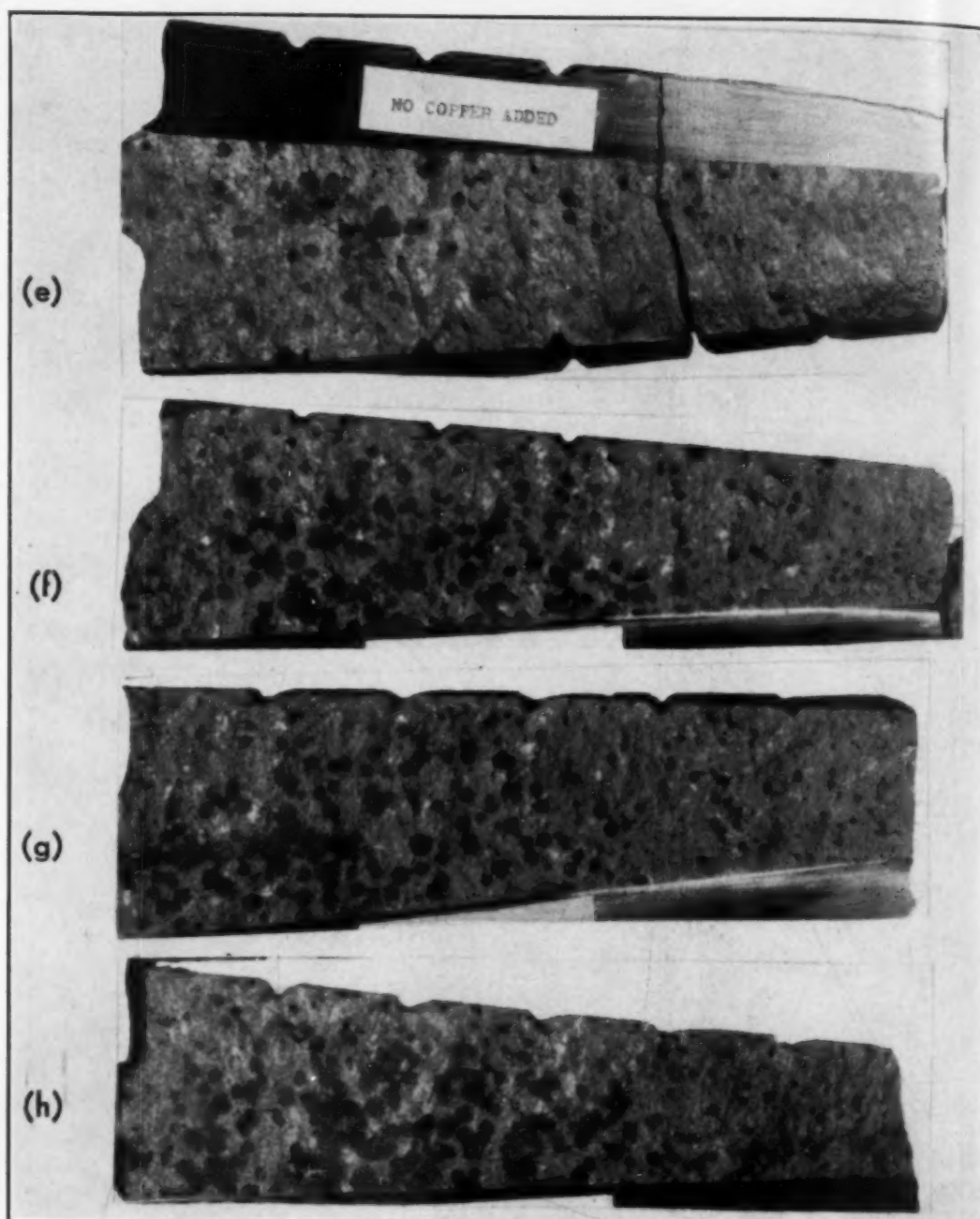


Fig. 19 (Continued)

(e) Standard Iron  
(g) 0.50% Cu Added  
2.77% C

1.05% Si

(f) 0.25% Cu Added  
(h) 1.00% Cu Added  
0.34% Mn

it is preferred for production purposes. With an iron like the B iron, there is no need to add copper for control of temper carbon spots because the addition of copper causes no further refinement in temper carbon size. The effect of copper upon annealing parallels its effect upon temper carbon distribution.

The microstructure of annealed samples is shown in Fig. 18. The iron made with Pig Iron A heated rapidly to 1700 degrees Fahr. shows large quantities of massive cementite after 8 hours at 1700 degrees Fahr. (925 degrees Cent.) but 1.00 per cent copper added to this iron results in practically complete elimination of carbides in 8 hours. The addition of copper to the B iron has very little effect upon speed of carbide elimination because the temper carbon spots are close packed even without the copper and first stage annealing is complete in 8 hours after rapid heating to 1700 degrees Fahr. (925 degrees Cent.). A small difference in amount of carbide existed after 3 hours at 1700 degrees Fahr. (925 degrees Cent.) in the rapidly heated samples but the difference is insignificant and of no commercial value.

When heating to 1700 degrees Fahr. (925 degrees Cent.) is slow the rate of carbide elimination in the irons with and without copper is almost identical. Immediately upon attaining 1700 degrees Fahr. (925 degrees Cent.) the samples containing copper showed less carbide present but after 4 hours the first stage is completed even for the irons without copper so the addition of copper for accelerating the annealing process in this particular kind of iron would cost more than the advantage gained.

This description of the effect of copper upon the freezing and annealing of white iron has been given as a basis of comparison with the effect of copper oxide so that it in turn could be compared with the effect of iron oxide. The iron to which copper oxide was added was of the type which develops close packed temper carbon upon heating rapidly to 1700 degrees Fahr. (925 degrees Cent.) in the annealing cycle like Iron B, Fig. 16. Fig. 19 shows the effect of the copper oxide upon the freezing structure of the iron as disclosed by fractured pyramid castings. The increase in mottling with copper oxide additions was greater than occurred when copper was added to iron of similar composition and annealing behavior (see Fig. 15), but no coarsening effect upon temper carbon distribution occurred such as that caused by the addition of iron oxide. The increased mottling tendency above that which might be expected of copper additions may be due to the effect of the oxygen added in combination with the copper. We have seen that oxide increases the tendency to mottle therefore it is plausible to attribute the increased mottling to oxide in this case because iron or silicon is capable of reducing the copper oxide so the effect should be the same as when iron oxide



alone is added. However, the presence of the copper seems to inhibit the deleterious effect of oxide upon the annealing process for the temper carbon distribution remains fine, and even with 1.25 per cent copper oxide added, primary graphitization is complete in 8 hours after heating rapidly to 1700 degrees Fahr. (925 degrees

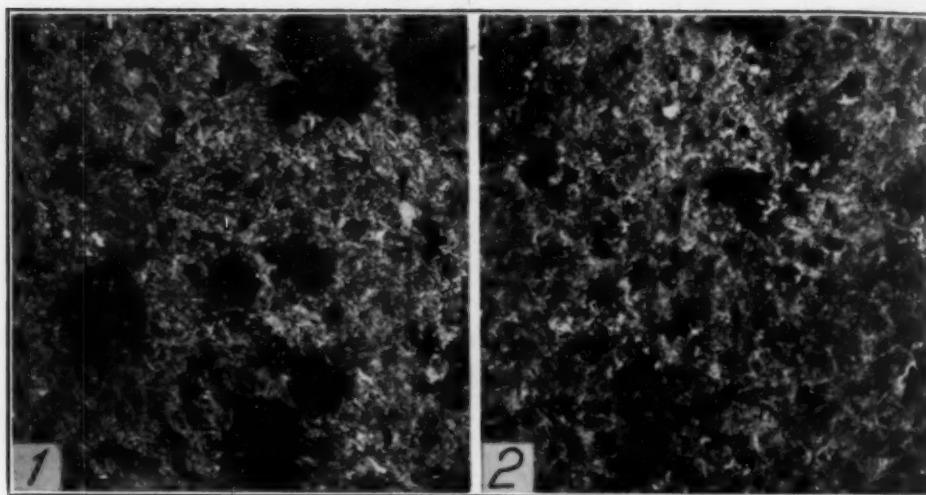


Fig. 20

Standard Iron—O  
2.75% C  
1.16% Si

Iron O Plus 1.00% Copper Oxide  
2.72% C  
1.06% Si

Cent.). As a matter of fact the oxygen made no difference at all in the annealing because the results obtained in the regular annealing cycle were the same as when similar copper additions without oxide were made. Upon reaching 1700 degrees Fahr. (925 degrees Cent.) the sample to which 0.50 per cent copper oxide had been added was considerably further advanced toward completion of first stage annealing than the base iron but in three hours at 1700 degrees Fahr. (925 degrees Cent.) this difference was erased and after completing the standard cycle the structures of all samples were identical. The possible use of copper suggested by this result is to counteract the deleterious effects of oxygen which may contaminate the iron in case of improper handling of the iron. However, prevention is better than cure and it has been demonstrated that similar results can be obtained without the use of copper so the use of copper for such purpose does not seem commercially justified. The control of the iron to obtain close packed temper carbon spots even when the annealing cycle includes rapid heating to 1700 degrees Fahr. (925 degrees Cent.) is done by means of selecting the silvery pig iron used for

12.5 per cent of the cupola charge. It is not definitely known what factor in the pig iron is responsible for this action but the desirable or undesirable characteristic can be obtained at will by merely switching pig irons.

If we were to completely analyze the influence of all the elements whether metallic, gaseous, or nonmetallic, upon solidification and upon annealing, we would find that not only would there be a considerable variation in their stabilizing power but that stabilizing power during solidification would not necessarily be associated with stabilizing power during annealing nor would graphitizing power of an element during solidification necessarily signify a similar power during annealing. Depending upon the mechanism by which the various elements operated to either graphitize or stabilize would be determined what their influence would be during either solidification or annealing. A hypothetical classification of the possible kinds of influence exerted by various elements during solidification and annealing might be something like that shown in the following:

Class	Carbide Retaining Power During Solidification	Carbide Stabilizing Power During Annealing	Example
1	Negative (strongly graphitizing)	Negative (strongly graphitizing)	C, Si, Cu and other graphitizers
2	Negative (graphitizing)	Weak	Oxygen added as $\text{Fe}_2\text{O}_3$ , $\text{Fe}_3\text{O}_4$
3	Weak	Weak	Manganese
4	Weak	Strong	Chromium
5	Strong	Strong	—
6	Strong	Weak	—
7	Strong	Neutral	Moisture

The mechanism by which these various results are obtained, whether by direct influence upon the iron carbide by combination with it, or by indirect effect by restricting its freedom of action, such as effected by nonmetallics in the melt, or by various concentrations in the solid affecting carbon migration and location of graphite precipitation, are all subjects for future detailed investigations. For those who have the necessary training and interest, these problems will provide as fascinating an investigation as can be found in any of the sciences.

## TAYLOR SPEED AND ITS RELATION TO REDUCTION OF AREA AND BRINELL HARDNESS

BY E. J. JANITZKY

### *Abstract*

*The ratio of Brinell hardness number to reduction of area as obtained in the usual tensile test gives an index of machinability for rough turning. By plotting this ratio against the Taylor speed a family of curves are obtained which express the relative machinability. Each curve expresses the change in machinability for steels of the same reduction of area as the Brinell hardness varies. The ratio of Brinell hardness to reduction of area is inversely proportional to the machinability as expressed by the Taylor speed.*

THE quest for an index of machinability began with Taylor's establishment of the conception that the measure of machinability may be expressed by the cutting speed at which a standard tool will last exactly 20 minutes.<sup>1</sup>

Taylor also found that the change in life of a high speed steel tool, resulting from a change in the cutting speed, could be represented approximately by an empirical equation:

$$VT^n = C \quad (1)$$

in which  $V$  = the cutting speed in feet per minute

$T$  = the tool life in minutes

$n$  = an exponent which Taylor found to be  $\frac{3}{8}$

$c$  = a constant which is dependent for its numerical value upon the exact cutting conditions (other than speed). It will vary with the form and size of tools, the material cut, the steel and treatment of the steel from which the tools are prepared, the feed and depth of cut.

Curves from which  $V$  is deduced are generally called TV curves.

Taylor also developed an empirical relation for carbon steels between cutting speed and the tensile properties of the steel tested by using the tensile stress and elongation. The equation gives some indication regarding machinability, but with the advent of alloy steels

<sup>1</sup>Fred W. Taylor, "On the Art of Cutting Metals," *Transactions, American Society of Mechanical Engineers*, Vol. 28, 1906.

The author, E. J. Janitzky, is consulting metallurgical engineer, Carnegie-Illinois Steel Corp., Chicago. Manuscript received Oct. 4, 1937.



it lost its usefulness. Taylor however was not satisfied with the evaluations by this equation.

The present tendency to judge machinability by tensile strength, yield point or Brinell hardness number separately is not satisfactory. Two steels with equal tensile strength or equal yield point may have different machining properties depending on the elastic ratio. Likewise two equal Brinell hardness numbers do not necessarily signify equal machinability.

Fig. 1 represents diagrams in which yield point, tensile strength,

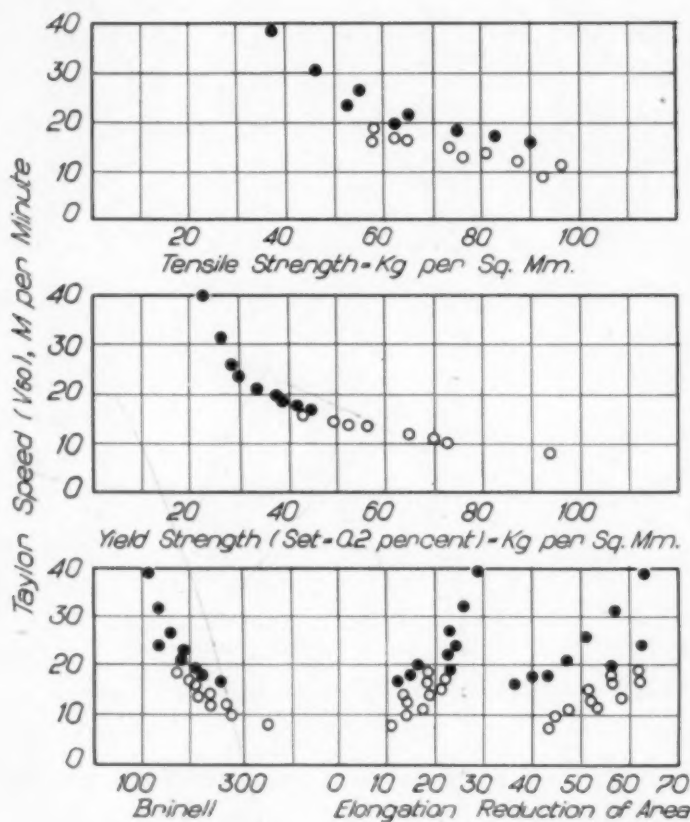


Fig. 1—Graphs Showing Yield Point, Tensile Strength, Reduction of Area, Elongation and Brinell Hardness Numbers Plotted Against Taylor Speed.

reduction of area, elongation and Brinell hardness number are each plotted independently against Taylor speed. These data were taken from F. Langenbach's<sup>2</sup> work given in Table I. In each plot, except for the yield point, the carbon steels separate themselves conspicuously from the alloy steels. (Carbon steels are denoted by black

<sup>2</sup>F. Langenbach, "Die Zerspanbarkeits-Kennziffer  $V_{100}$  in ihrer Beziehung zur Zugfestigkeit und Streckgrenze beim Schupp-Dreh Vorgang von Legierten und unlegierten Stählen." Dissertation, Aachen, 1932.

Table I  
Compilation of Data on Material under Taylor Test

No.	S.A.E. Grade	Heat Treatment in C°	Tensile		Yield Point		Elongation	Reduction of Area
			Kg.sq. mm.	1000 psi	Kg.sq. mm.	1000 psi		
1	1020	N 920°C 1688°F	37.5	53.3	22.0	31.3	29.3	62.0
2	1025	N 880°C T 700°C 1616°F 1292°F	46.3	65.8	26.3	37.4	26.6	57.0
3	1035	O 850°C T 720°C 1562°F 1328°F	55.0	78.2	28.0	39.8	23.6	51.0
4	1045	O 850°C T 720°C 1562°F 1328°F	64.7	92.0	34.0	48.3	23.2	47.0
5	1070	A 720°C 1328°F	75.4	107.0	39.5	56.2	16.2	43.0
6	1075	O 850°C T 650°C 1562°F 1202°F	83.5	118.0	43.2	61.4	14.5	40.0
7	1080	O 850°C T 650°C 1562°F 1202°F	90.0	127.9	45.7	64.9	13.2	37.0
8	X-1020	W 820°C T 650°C 1508°F 1202°F	51.5	73.2	30.5	43.4	25.6	62.0
9	X-1030	W 820°C T 650°C 1508°F 1202°F	63.1	89.7	38.8	55.2	23.2	56.0
10	T-1340	O 850°C T 680°C 1562°F 1256°F	74.2	105.5	50.5	71.8	21.3	52.0
11	2320	O 850°C T 670°C 1562°F 1238°F	58.6	83.3	39.2	55.7	23.8	62.0
12	2520	O 850°C T 670°C 1562°F 1238°F	61.8	87.9	41.7	59.3	22.0	63.0
13	2330	O 830°C T 500°C 1526°F 932°F	65.6	93.3	45.0	63.9	19.3	56.0
14	5130	O 840°C T 550°C 1544°F 1022°F	81.5	115.9	53.0	75.4	14.5	52.0
15	5140	O 840°C T 550°C 1544°F 1022°F	87.5	124.4	65.6	93.3	15.2	53.0
16	5160	O 840°C T 550°C 1544°F 1022°F	96.6	136.5	70.3	100.4	12.8	48.0
17	3230	O 850°C T 670°C 1562°F 1238°F	58.5	83.2	42.0	59.7	19.3	56.0
18	3330	O 820°C T 680°C 1508°F 1256°F	76.3	108.5	56.5	80.3	20.0	58.0
19	3340	O 820°C T 620°C 1508°F 1148°F	93.4	132.8	72.5	102.1	15.6	45.0
20	5% Ni-1.5 Cr	O 820°C T 550°C 1508°F 1022°F	118.5	168.5	94.0	133.7	11.5	44.0

N = Normalized—A = Annealed—O = Oil Quenched—W = Water Quenched—T = Tempered.

circles.) Since these tests were made near the surface of large quenched and tempered bars (6 to 8 inches in diameter), this spread in tensile properties may be ascribed to the difference in hardenability in large section of the plain carbon steel as compared to the alloy steels tested. The excellent correlation of yield point with Taylor speed is regarded as due to the selection of steels, since Langenbach did not test any alloy steels annealed to give a low yield point. It is known from experience that annealed alloy steels generally machine with greater difficulty than plain carbon steels of the same yield point. It will also be recognized from experience that the relation of yield point to Taylor speed in Fig. 1 also fails in the case of an austenitic alloy such as 18 per cent chromium, 8 per cent

Table 1—(Continued)  
Compilation of Data on Material under Taylor Test

No.	S.A.E. Grade	Heat Treatment in C°	Brinell	Actual V <sub>60</sub> m/min.	Calcul. V <sub>60</sub> m/min.	Actual V <sub>60</sub> f/min.	Calcul. V <sub>60</sub> f/min.
1	1020	N 920°C 1688°F	111	39.5	38.6	129.2	127.0
2	1025	N 880°C 1616°F	127	31.5	33.7	103.9	109.0
3	1035	O 850°C 1562°F	156	26.5	27.1	86.6	86.3
4	1045	O 850°C 1562°F	182	21.5	22.8	70.3	74.5
5	1070	A 720°C 1328°F	209	19.0	19.9	63.1	65.0
6	1075	O 850°C 1562°F	234	18.0	17.9	58.8	58.5
7	1080	O 850°C 1562°F	255	16.5	16.7	53.9	54.6
8	X-1020	W 820°C 1508°F	145	24.0	25.0	78.5	81.7
9	X-1030	W 820°C 1508°F	180	20.0	19.4	65.4	63.5
10	T-1340	O 850°C 1562°F	209	15.0	16.4	49.0	53.6
11	2320	O 850°C 1562°F	174	19.0	18.6	65.1	60.8
12	2520	O 850°C 1562°F	187	17.5	16.3	57.2	53.3
13	2330	O 830°C 1526°F	195	17.0	17.1	56.0	55.9
14	5130	O 840°C 1544°F	236	14.0	13.5	45.8	44.1
15	5140	O 840°C 1544°F	255	12.5	11.9	40.8	39.0
16	5160	O 840°C 1544°F	278	11.5	11.2	37.6	36.6
17	3230	O 850°C 1562°F	174	18.5	20.6	60.5	67.3
18	3330	O 820°C 1508°F	224	14.0	13.1	45.0	42.8
19	3340	O 820°C 1508°F	282	10.0	11.6	32.7	37.9
20	5% Ni-1.5 Cr	O 820°C 1508°F	354	7.5	8.2	24.4	26.8

N = Normalized—A = Annealed—O = Oil Quenched—W = Water Quenched—  
T = Tempered.

nickel, which has a low yield point but is known to machine with great difficulty. The latter point was well recognized by Langenbach himself.

The ambiguity resulting in the plots of single physical properties against Taylor speed suggests an attack by means of a family of curves and the establishment of an interrelation of the curves among each other. With this idea in mind the writer plotted the ratio of Brinell hardness number to reduction of area against Taylor speed, noting for each point so obtained the numerical value of the corresponding per cent reduction of area.

The data of F. Langenbach<sup>2</sup> were selected for the development of a general equation for such a family of curves for calculating Taylor speed from the per cent reduction of area and Brinell num-



ber. These data include plain carbon, medium and high manganese, nickel, chromium, and chromium-nickel steels. The data of Langenbach were selected not only because of the wide range of analyses represented, but also because of precautions used in taking the tests, and the fact that only a limited zone was used for making both the machinability tests and the tests for physical properties. As the material under test (bars from 6 to 8 inches in diameter and from 60 to 120 inches long) was in the heat treated condition, it is not immaterial how deep machining is performed in regard to the diameter or the location of the tests taken for tensility and Brinell hardness. Fortunately, the machining depth was limited to 2 inches maxi-

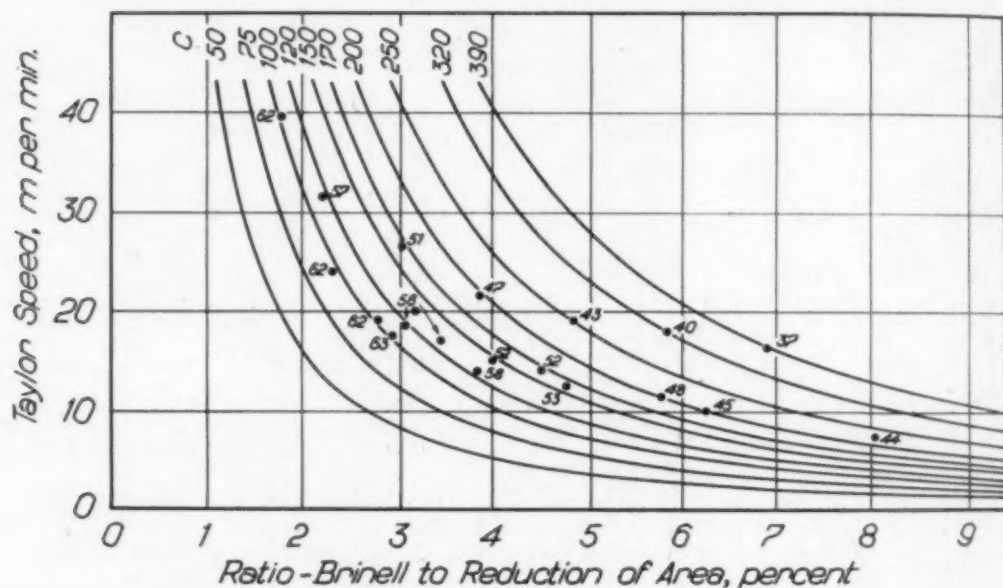


Fig. 2—Curves Showing Ratio of Brinell Hardness Numbers to Per Cent Reduction of Area Plotted Against Taylor Speed.

mum in order to avoid discrepancies on account of hardness gradients and the tensile test specimens were taken longitudinally, close to the surface at the ends of the rounds (gage length = 5 times the diameter).

The machining was of the roughing type. The dimensions of the tool are given as:

cross section of tool	= 1.18 x 1.574 inches
front clearance	= 6 degrees
lip angle	= 65 degrees
back slope	= 19 degrees
adjusting angle	= 35 degrees
tool point angle	= 80 degrees

The recorded data on machinability represent a depth of cut of 0.236

inch and a feed of 0.039 inch per revolution. The Taylor speed,  $V_{60}$ , was obtained by Taylor's method from the TV curves. Each of the TV curves was duplicated and the data so obtained were averaged. If the duplicate tests were not in agreement a triplicate test was run. It is perhaps not out of place to mention that the object of these tests was to establish whether there was a difference between tipped tools as against standard tools. Thus the machinability of each steel was determined in duplicate with tipped tools and in duplicate with standard tools. No difference was observed between tipped and standard tools.

As mentioned, the ratio of Brinell hardness number to per cent reduction of area was plotted against the Taylor speed, noting beside each point so obtained the numerical value of the per cent reduction of area for that particular test. By this procedure it was observed (see Fig. 2) that the points for tests with equal per cent reduction of area aligned themselves around hyperbolic curves which may be expressed by the following equation:

$$V_{60} = \frac{c}{\left(\frac{B}{R}\right)^n} \quad (2)$$

in which  $V_{60}$  = Taylor speed (as in equation (1) for 60 minutes tool life)  
 $c$  = a constant (different for each equal reduction of area curve)  
 $B$  = Brinell hardness number  
 $R$  = per cent reduction of area  
 $n$  = an exponent which is constant for all the curves and was found from the experimental data to equal 1.63.

Since the exponent is constant for each of the equal reduction of area curves, it is possible to establish an interrelation for the whole family of equal reduction of area curves. For this purpose the constant  $c$ , for each equal reduction of area curve, was plotted against the per cent reduction of area, (see Fig. 3). The curve so obtained was also hyperbolic and may be expressed by the equation:

$$cR^m = C \quad (3)$$

in which  $c$  and  $R$  have the same meaning as in equation (2)

and  $C$  = a constant = 5,390,000 when  $V$  is expressed in meters; when  
 $V$  is expressed in feet  $C$  = 17,625,300  
 $m$  = an exponent = 2.64

solving for  $c$  and substituting in equation (2), we obtain

$$V_{60} = \frac{C}{B^n R^{(m-n)}} \quad (4)$$

Therefore the equation for  $V_{60}$  for cutting depth of 0.236 and a feed of 0.039 inch per revolution may be written

$$V_{60} = \frac{C}{B^{1.63} R^{1.01}} \quad (4a)$$

The constant  $C$  depends for its numerical value upon the exact cutting conditions (other than speed). It will vary with the form and size of the tool used, the steel and its heat treatment from which the tools are made, as well as the feed and depth of cut used.

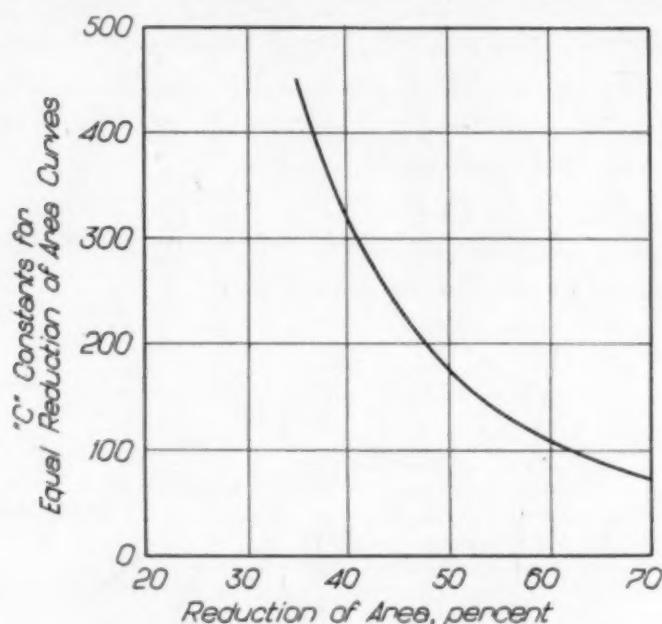


Fig. 3—Curve Showing Per Cent Reduction of Area Plotted Against Constants for Equal Reduction of Area Curves.

Langenbach's experimental data together with the data calculated by equation (4a) are given in Table I. The calculated Taylor speeds conform closely to the experimental data. A separation of steels according to their hardenabilities is not noticeable.

That the equation herein presented is applicable to a wider range of steels than the somewhat limited group studied by Langenbach may be demonstrated by the case of an austenitic steel, 18 per cent chromium and 8 per cent nickel, of the following physical properties:

Yield point 24.5 kilograms per square millimeter	= 35,000 pounds per sq. in.
Reduction of Area	= 73.1 per cent
Brinell number	= 207

According to Fig. 1 using yield point, the Taylor speed,  $V_{60}$ , should



be 31 meters per minute (103.9 feet per minute). This would make the 18-8 alloy as easily machinable as 1020 as shown in Table I which we know from experience is not the case. However, by using equation 4a, a Taylor speed of 10.6 meters per minute (34.6 feet per minute) is obtained which is in accordance with estimations on hand. Furthermore it will be recognized from experience that this 18-8 alloy machines with about the same difficulty as 3340 (as heat treated in Table I) which was determined by Langenbach to have a Taylor speed of 10.0 meters per minute.

It will be noted by comparison of calculated and actual Taylor speed (Table I), that variation in chemical composition does not influence machinability provided that the steels may be heat treated to have the same mechanical properties, namely Brinell number and per cent reduction of area. In other words if two steels are heat treated to have the same mechanical properties, assuming such heat treatment is possible, they will be equal in regard to machinability.

For convenience, indices relative to machinability based on equation 4a were worked out and are given in Table II. The author is indebted to Dr. Asimow of the Carnegie-Illinois Steel Corporation for the indices given in the table. These are derived from the equation 4a with the constant adjusted to give a value for  $V_{60}$  of 100 for the arbitrary combination of Brinell hardness number equal to 100 and the reduction of area equal to 30 per cent. Other steels having higher physical properties can therefore be compared with this arbitrary combination as a base. If one steel has an index of 50 and another has an index of 25 then for the same cutting conditions, to obtain a tool life of 1 hour the steels must be machined respectively at speeds  $\frac{1}{2}$  and  $\frac{1}{4}$  of the speed for the arbitrary base. In other words, for a tool life of 1 hour the first steel may be machined at twice the speed for the second steel. Hence by means of the indices in the table the various steels can be given relative machinability ratings.

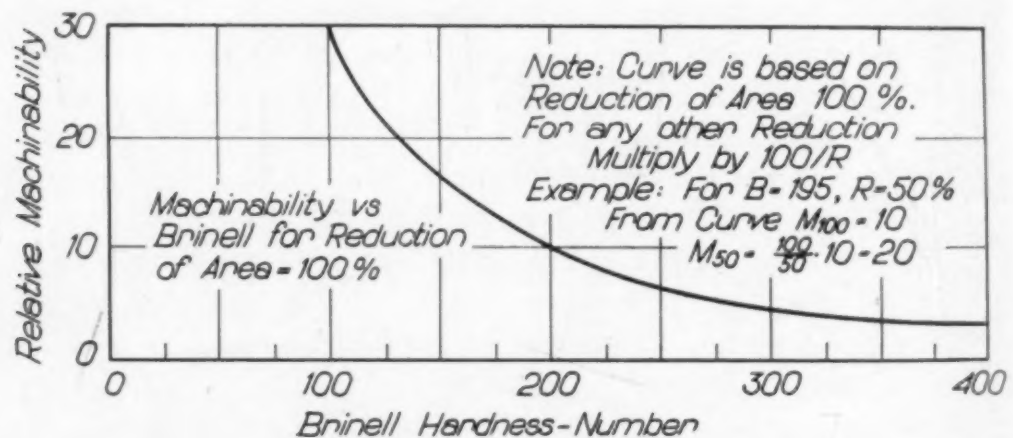
For any given set of cutting conditions the actual speeds can be calculated as soon as a suitable factor is determined by experiment. Thus for the conditions of Langenbach's experiments the factor is 3.13. If the indices are multiplied by this factor the resulting values correspond to the speeds as determined in his experiments.

If, in the equation 4a the exponent of the per cent reduction in area is assumed to be 1.00 instead of 1.01 then it is possible to reduce the formula to a single curve based on a reduction of area of 100

**Table II**  
**Relative Machinability Numbers for Steels**

Brinell Hdn. No.	(Based on E. J. Janitzky's Formula for a Taylor Speed $V_{60}$ ) Per Cent Reduction of Area							
	30	35	40	45	50	55	60	65
100	100.0	85.7	74.9	66.6	59.8	54.3	49.7	45.9
110	85.6	73.4	64.1	57.0	51.7	46.5	42.5	39.3
120	74.4	63.7	55.7	49.5	44.5	40.4	37.0	34.2
130	65.0	55.7	48.6	43.3	38.9	35.3	32.3	29.8
140	57.8	49.6	43.2	38.5	34.6	31.4	28.7	26.5
150	51.8	44.5	38.8	34.6	31.0	28.2	25.8	23.8
160	46.7	40.1	35.0	31.2	27.9	25.4	23.2	21.4
170	42.0	36.0	31.4	28.0	25.1	22.8	20.9	19.2
180	38.4	32.9	28.7	25.6	22.9	20.8	19.1	17.6
190	35.2	30.2	26.3	23.5	21.0	19.1	17.5	16.1
200	32.3	27.7	24.2	21.5	19.3	17.5	16.1	14.8
210	29.7	25.5	22.2	19.8	17.7	16.1	14.8	13.6
220	27.5	23.6	20.6	18.3	16.4	15.0	13.7	12.6
230	25.7	22.0	19.2	17.1	15.4	14.0	12.8	11.8
240	24.1	20.7	18.0	16.0	14.4	13.1	12.0	11.0
250	22.5	19.3	16.9	15.0	13.5	12.2	11.2	10.3
260	21.0	18.0	15.7	14.0	12.5	11.4	10.3	9.6
270	19.7	16.9	14.7	13.1	11.8	10.7	9.8	9.0
280	18.7	16.0	14.0	12.5	11.2	10.2	9.3	8.6
290	17.7	15.2	13.2	11.8	10.6	9.6	8.8	8.1
300	16.7	14.3	12.5	11.1	10.0	9.1	8.3	7.7
310	15.9	13.6	11.9	10.6	9.5	8.6	7.9	7.3
320	15.1	13.0	11.3	10.1	9.0	8.2	7.5	6.9
330	14.4	12.3	10.8	9.6	8.6	7.8	7.2	6.6
340	13.7	11.7	10.3	9.1	8.2	7.4	6.8	6.3
350	13.0	11.1	9.7	8.7	7.8	7.1	6.5	6.0
360	12.4	10.6	9.3	8.3	7.4	6.7	6.2	5.7
370	11.8	10.2	8.8	7.9	7.1	6.4	5.9	5.4
380	11.3	9.7	8.5	7.5	6.8	6.1	5.6	5.2
390	10.9	9.3	8.2	7.2	6.5	5.9	5.4	5.0
400	10.5	9.0	7.9	7.0	6.3	5.7	5.2	4.8

Example for Table: Factor = 3.13 (60 min. tool life). For dry roughing cut: depth—.236", feed—.039 in/rev., Frt. clearance—6°, back rake—19°.



per cent, from which can be readily derived the values for the entire family of curves as shown in Fig. 2. For a steel of any Brinell hardness number, for example  $B = 195$ , a relative machinability number is obtained from the curve. In this illustration the value from the curve is 10. This is the machinability rating for a hypothetical steel having a Brinell hardness number of 195 and a reduc-

tion of area of 100 per cent. For the actual steel which has, say, a reduction of area equal to 50 per cent, the rating is the machinability number

$$M_{50} = M_{100} \times \frac{100 \text{ per cent}}{50 \text{ per cent}}$$

$$= 10 \times \frac{100}{50} = 20$$

### SUMMARY

To summarize briefly, it has been possible by use of the Brinell hardness and the reduction of area as determined in the usual tensile test to obtain an index of machinability for rough turning. The relation between Taylor speed and the ratio, Brinell hardness/reduction of area, has been expressed mathematically. A graphical presentation has also been given. The use of the ratio, Brinell hardness/reduction of area, allows treatment of plain carbon steels, alloy steels, and austenitic steels by the same methods, a distinct advantage over the use of any one of the four components of the tensile test alone.



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TECHNICAL PROGRAM AND REPORTS OF OFFICERS  
AMERICAN SOCIETY FOR METALS—20th ANNUAL  
CONVENTION, DETROIT, OCTOBER 17 to 21, 1938

FOR the benefit of members who were not in attendance at the Twentieth Annual Convention of the Society, held in Detroit, October 17 to 21, 1938, and for purposes of record, the Technical Papers Program and Reports of Officers presented at the annual meeting are herewith published in full.

MONDAY, OCTOBER 17

Morning Session—Grand Ballroom, Statler Hotel

Joint Chairmen—J. M. Watson and W. P. Woodside

*Softening Rate of a Steel When Tempered from Different Initial Structures*, by E. H. Engel, Carnegie-Illinois Steel Corp., Vandergrift, Pa.

*Selective Hardening with the Oxy-Acetylene Flame*, by R. L. Rolf, Lakeside Steel Improvement Co., Cleveland.

*Reactions to Annealing above the Eutectoid Temperature of Quenched Hyper-eutectoid Steels*, by C. R. Austin and M. C. Fetzer, Pennsylvania State College.

Morning Simultaneous Session—Michigan Room, Statler Hotel

Joint Chairmen—H. A. Anderson and W. H. Swanger

*Relation Between the Rockwell "C" and Diamond Pyramid Hardness Scales*, by Howard Scott and T. H. Gray, Westinghouse Electric & Mfg. Co., E. Pittsburgh.

*Absolute Hardness*, by E. G. Mahin and G. J. Foss, Jr., University of Notre Dame.

*The Effects of Fibre on the Notch Toughness of Mild Steel as Influenced by Cooling Rate*, by O. W. Ellis, Ontario Research Foundation, Toronto, and J. E. McDonell, Queen's University, Kingston.

*A New Method for the Rapid Determination of Carbon in Samples of Plain Carbon Open-Hearth Steel*, by B. A. Rogers, Karl Wentzel and J. P. Riott, Bureau of Mines, Pittsburgh.

Afternoon Session—Convention Hall

Joint Chairmen—R. W. Schlumpf and N. I. Stotz

*Effects of Special Alloy Additions to Stainless Steels*, by Russell Franks, Union Carbide & Carbon Research Laboratories, Inc., Niagara Falls.

*Stress-Corrosion Cracking in Austenitic Stainless Steels*, by S. L. Hoyt and M. A. Scheil, A. O. Smith Corp., Milwaukee.

*Some Observations on the Passivity of Stainless Steels*, by J. N. Ostrofsky, Rustless Iron and Steel Corp., Baltimore.

Convention Hall

Educational Lectures

*Physics of Metal Cutting*, by Hans Ernst, Cincinnati Milling Machine and Cincinnati Grinders, Inc., Cincinnati.

*Scientific Foundations of the Pyrometry of Solids*, by R. B. Sosman, U. S. Steel Corp., Kearny, N. J.

TUESDAY, OCTOBER 18

Morning Session—Grand Ballroom, Statler Hotel

Joint Chairmen—H. J. French and J. P. Gill

*Influence of Chromium, Silicon and Aluminum on the Oxidation Resistance of Intermediate Alloy Steels*, by A. E. White, C. L. Clark, University of Michigan, and C. H. McCollam, Timken Roller Bearing Co., Canton.

*Influence of Aluminum on Cast Corrosion Resisting Steel*, by J. A. Duma, Norfolk Navy Yard, Portsmouth, Va.

*Development in Molybdenum High Speed Cutting Steels*, by W. R. Breeler, Allegheny Ludlum Steel Corp., Dunkirk, N. Y.

**Morning Simultaneous Session—Michigan Room, Statler Hotel**

Joint Chairmen—W. P. Eddy, Jr. and L. W. Kempf

*Some Properties of Oxygen-Free High Conductivity Copper (OFHC)*, by C. G. Goetzel, Hardy Metallurgical Co., New York.*A New 70/30 Nickel-Copper Alloy Subject to Precipitation Hardening*, by Erich Fetz, Wilbur B. Driver Co., Newark.*Effects of Substantial Additions of Aluminum, Cobalt, Titanium and Columbium on the Properties of 80 Ni-20 Cr Alloys*, by A. L. Sanford and O. E. Harder, Battelle Memorial Institute, Columbus, O.**Afternoon Session—Convention Hall**

Joint Chairmen—John Chipman and H. W. McQuaid

*The Production of Flakes by Treating Molten Steel with Hydrogen and the Time of Cooling Necessary to Prevent Their Formation*, by R. E. Cramer and E. C. Bast, University of Illinois.*Effect of Carbon Content on the Oxidation of Steel in Air at High Temperatures*, by C. A. Siebert, University of Michigan.*Some Effects of Deoxidizers in Low Carbon, 1.5 Per Cent Chromium Steel*, by Walter Crafts and J. L. Lamont, Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls.**Convention Hall***Educational Lectures**Machinability of Ingot Iron, Wrought Iron, S.A.E. Steels and Stainless Steels*, by H. B. Knowlton, International Harvester Co., Chicago.*The Pyrometry of Metals by Means of Thermocouples*, by R. B. Sosman, U. S. Steel Corp., Kearny, N. J.**WEDNESDAY, OCTOBER 19****Morning Session—Grand Ballroom, Statler Hotel****ANNUAL MEETING OF THE A.S.M.**

1938 Edward de Mille Campbell Memorial Lecture presented by Alfred L. Boegehold, General Motors Research Laboratories, Detroit.

Chairman—Dr. George B. Waterhouse

**Afternoon Session—Convention Hall**

Joint Chairmen—O. W. Ellis and M. F. Judkins

*Graphitizing Rate and Nodule Number*, by H. A. Schwartz and M. K. Barnett, National Malleable & Steel Castings Co., Cleveland.*The Effect of Type of Cold Deformation on the Recrystallization Properties of Armco Iron*, by H. F. Kaiser and H. F. Taylor, Naval Research Laboratory, Washington.*Constitution Diagrams for Iron-Carbon-Molybdenum Alloys*, by J. R. Blanchard, R. M. Parke, and A. J. Herzig, Climax Molybdenum Co., Detroit.**Convention Hall***Educational Lectures**Some Factors Affecting the Machinability of Cast Steel, Cast Iron, and Malleable Iron*, by J. W. Bolton, Lunkenheimer Co., Cincinnati.*The Optical and Radiation Pyrometry of Solids and Surfaces*, by R. B. Sosman, U. S. Steel Corp., Kearny, N. J.**THURSDAY, OCTOBER 20****Morning Session—Grand Ballroom, Statler Hotel**

Joint Chairmen—L. S. Bergen and J. L. Burns

*White Layer Structure in the Erosion of Machine Gun Barrels*, by W. H. Snair, American Can Co., Maywood, Ill., and W. P. Wood, University of Mich.*The Influence of Microstructure upon the Process of Diffusion in Solid Metals*, by F. N. Rhines and Cyril Wells, Carnegie Institute of Technology.*Study of Carbide Solution in Hypoeutectoid Plain Carbon and Low Alloy Commercial Steels*, by R. H. Lauderdale and O. E. Harder, Battelle Memorial Institute, Columbus, O.

**Simultaneous Morning Session—Michigan Room, Statler Hotel**

Joint Chairmen—R. L. Dowdell and R. L. Wilson

*Possible Uses of Radioactive Substances in the Testing of Metals*, by Herman F. Kaiser, Naval Research Laboratory, Washington.*Color Carbon and Aging*, by Carl L. Shapiro, Consulting Engineer, Syracuse, N. Y.*Hardness Gradients in Tempered Steel Cylinders*, by C. A. Rowe and R. A. Ragatz, University of Wisconsin.**Afternoon Session—Convention Hall***Symposium on Hardenability of Medium and Low Alloy Steels*

Joint Chairmen—E. C. Bain and A. A. Bates

*The Physics of Hardenability. The Mechanism and the Rate of the Decomposition of Austenite*, by R. F. Mehl, Carnegie Institute of Technology.*Hardenability Tests*, by W. E. Jominy, General Motors Corp., Detroit.*The Effect of the Silicon and Aluminum Addition on the Hardenability of Commercial Steels*, by M. J. R. Morris and H. W. McQuaid, Republic Steel Corp., Massillon and Cleveland.**Convention Hall***Educational Lecture**Machinability of Tool Steels*, by A. H. d'Arcambal and W. E. Bancroft, Pratt and Whitney Co., Hartford.**FRIDAY, OCTOBER 21****Morning Session—Grand Ballroom, Statler Hotel***Symposium on Hardenability of Medium and Low Alloy Steels*

Joint Chairmen—R. S. Archer and Bradley Stoughton

*Hardenability, Its Relation to Quenching and Some Quantitative Data*, by M. A. Grossmann, M. Asimow and S. F. Urban, Carnegie-Illinois Steel Corp., Chicago.*Hardenability of Low Chromium Steels*, by Walter Crafts and J. L. Lamont, Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls.*Transverse Hardness Tests of Heat Treated Steels*, by G. T. Williams, Cleveland Tractor Co., Cleveland.**Morning Simultaneous Session—Michigan Room, Statler Hotel**

Joint Chairmen—O. E. Harder and E. G. Mahin

*A Method for the Preparation of Metallographic Specimens*, by G. A. Ellinger and J. S. Acken, National Bureau of Standards.*Automatic Polishing of Metallurgical Specimens Using Cast Iron and Lead Laps*, by T. C. Jarrett, American Optical Co., Southbridge, Mass.*A Metallographic Approach to the Study of the Sensitivity of Steel to Cold Work*, by H. K. Work and S. L. Case, Jones and Laughlin Steel Corp., Pittsburgh.**Afternoon Session—Convention Hall***Symposium on Hardenability of Medium and Low Alloy Steels*

Joint Chairmen—S. L. Hoyt and E. S. Davenport

*Hardenability and its Designation, the Hardenability Line*, by B. R. Queneau, Columbia University, and W. H. Mayo, Carnegie-Illinois Steel Corp., Homestead, Pa.*Hardenability of Plain Carbon Steels*, by J. L. Burns, Republic Steel Corp., Chicago, and G. C. Riegel, Caterpillar Tractor Co., Peoria.*Hardenability in Light Sections*, by G. V. Luerssen, Carpenter Steel Co., Reading, Pa.**Convention Hall***Educational Lecture**Machining of Nonferrous Materials*, by H. P. Croft, Chase Brass and Copper Co., Cleveland.



**ANNUAL ADDRESS OF THE PRESIDENT***Twentieth Annual Convention, Detroit, October 19, 1938**GEORGE B. WATERHOUSE, President*

IT is a great pleasure to report that the Society is in good condition and has had a very successful year from every standpoint. Notwithstanding the depressed state of business during the year our membership has kept up, and the report of the Treasurer will show that we are in splendid financial condition. This is largely due to continued interest in the work of the Society on the part of the members, and is evidenced by the successful Chapter activities and the work of the various committees.

The Board of Trustees held four meetings during the year: November 19, 1937; March 22, 1938; August 19, 1938 and October 18, 1938. The reports of these meetings are published in *THE REVIEW* and show the conscientious and earnest efforts of your Trustees to serve properly the Society.

At the August meeting, due to the selection of James P. Gill, a member of the board of trustees for the office of vice president, the resignation of Mr. Gill was received by the board of trustees and accepted.

To fill Mr. Gill's unexpired term, the board of trustees, according to the privileges vested in them by the constitution of the Society, selected Dr. Samuel L. Hoyt of the A. O. Smith Corp., Milwaukee, to be a trustee of the Society for a period of one year to fill this unexpired term.

The President's Medal was presented to Past President Robert S. Archer at the Annual Banquet at Atlantic City, October 21, 1937.

The Henry Marion Howe Medal awarded to the paper of highest merit published in *TRANSACTIONS* during the year was awarded to Dr. Eric R. Jette of Columbia University and Mr. A. G. H. Anderson for their paper entitled "X-Ray Investigation of the Iron-Chromium-Silicon Phase Diagram" and was presented at the Annual Banquet, Atlantic City, October 21, 1937.

The Campbell Memorial Lecture established in 1925 and presented each year at the close of the Annual Meeting is eagerly awaited and always proves to be of importance and interest. The 1937 lecture was no exception to this rule and was presented by Dr. W. P. Sykes, Research Metallurgist of the General Electric Company, Cleveland, on "Structural and Hardening Characteristics of some Iron-Cobalt-

Tungsten Alloys." It was published in full in the *TRANSACTIONS*.

The Sauveur Achievement Award, which was established in 1934 and which is awarded by the Past Presidents of the Society acting as a Committee, was not given in 1937, due to a change made by the Committee in the rules for reaching a decision. An award will be made in 1938 to be announced at the Annual Banquet.

It had been expected that the Iron and Steel Institute and the Institute of Metals, both of Great Britain, would hold a joint meeting in this country in October, and they had been invited to be present at Detroit. Unfortunately their visit had to be cancelled, although a number of individual members have accepted our invitation.

Brief mention may be made of the work of some of the National committees of the Society:

The Handbook Committee is hard at work on the new edition of the Metals Handbook, thoroughly revised and largely rewritten. Our last edition was exhausted some time ago, well ahead of the expected date. Its publication is expected about April 1, 1939. The Society is grateful to the many members who work on the main committee and the numerous subcommittees.

An Education Committee was authorized during the year as a National Committee, in keeping with the importance of our Educational work. The committee has been active in many ways, particularly in the field of Visual Education. This Committee and the Headquarters Staff have been exceedingly helpful to the Chapters in connection with educational activities. The Committee has also arranged the very successful series of lectures this week, namely the evening lectures on "Pyrometry" by Dr. R. B. Sosman, United States Steel Corporation Research Laboratories; and the afternoon lectures on "Machinability." These afternoon lectures and their subjects are: "Physics of Cutting," Hans Ernst, Cincinnati Milling Machine Company; "Machinability of Ingot Iron, Wrought Iron, S. A. E. Steels and Stainless Steels," H. B. Knowlton, International Harvester Company; "Some Factors Affecting the Machinability of Cast Steel, Cast Iron and Malleable Iron," John W. Bolton, Lunkenheimer Company; "Machinability of Tool Steels," A. H. d'Arcambal, Past President, and W. E. Bancroft, Pratt and Whitney Company; "Machining Non-ferrous Materials (Cast and Wrought)," H. P. Croft, Chase Brass and Copper Company.

The thanks of the Trustees and the Society are due these lecturers for their splendid work.

Finally, opportunity must be taken to thank sincerely the technical societies working with us so whole-heartedly to make this Congress and Exposition a success, the American Welding Society, the Wire Association, and the Institute of Metals and Iron and Steel Divisions of the American Institute of Mining and Metallurgical Engineers.

#### *Award of President's Bell*

It has been a great problem to select the Chapter to which should be awarded the President's Bell. This award was instituted in 1926 to be made to Chapters for outstanding services to its members and the Society. It has been a great pleasure to visit more than half of the forty-eight Chapters this year, to talk with the officers and to take part in the meetings. All the Chapters are doing excellent work. The meetings are well planned and conducted, the speakers are carefully chosen, discussion is active, and in particular great attention is being paid to the educational activities. The award this year is given to the Los Angeles Chapter with Cincinnati a close second. Los Angeles is chosen because of the good work being done, and its outstanding services in connection with the Western Metal Congress and Exposition so successfully held during the week of March 21, 1938.

### REPORT OF THE TREASURER

BRADLEY STOUGHTON, *Treasurer*

The Society has completed on August 31, 1938, another very successful year, financially, and it is a pleasure for the treasurer to submit this report.

A larger cash balance and better cash position are shown on the August 31, 1938, statement than ever before, this total being \$70,677.89 against a total of \$51,306.83 on August 31, 1937, an increase of approximately 38 per cent and security holdings at the same time have increased 18 per cent and total assets 12.6 per cent. The only asset to decrease in total was accounts receivable, which indicates good collections and a continued healthy condition of accounts. Inventories remained approximately the same, additions of new items such as membership pins, membership certificate frames, automobile emblems and several new books offsetting a low inventory of paper and cover stock for the magazines. This paper and cover stock inventory has been replenished after the books were closed.



The Finance Committee and Board of Trustees have given very careful consideration and a great deal of time and thought to investment problems during the past year. Market conditions have been unsettled and no investments were decided upon until August 1938 when it was decided to invest in the following securities:

*Stocks*

100 shares General Motors Corp.  
 100 shares Kennecott Copper Corp.  
 50 shares International Nickel of Canada  
 50 shares Standard Oil Co. of New Jersey  
 50 shares Sears Roebuck & Co.

*Bonds*

\$5000.00 P. V. Idaho Power Co. 3¾'s  
 5000.00 P. V. Bethlehem Steel Corp. 4¼'s  
 5000.00 P. V. Southern Bell Telephone & Telegraph 3¼'s  
 5000.00 P. V. Goodyear Tire & Rubber Co. 5's  
 5000.00 P. V. Skelley Oil Co. 4's  
 5000.00 P. V. U. S. Steel Corp. 3¼'s  
 5000.00 P. V. Phillips Petroleum Co. 3's  
 5000.00 P. V. Phelps Dodge Corp. 3¼'s

The entire list had not been purchased at the close of business August 31 but \$34,000.00 worth had been added to the list with commitments made for the balance of approximately \$24,000.00. These investments are of the highest grade and add considerable strength to the portfolio. The stocks purchased provide an additional hedge against possible inflation. The portfolio when completed will show the following:

	Per Cent
Government Securities .....	22.7
Utilities .....	22.7
Railroads .....	14.8
Industrials .....	20.6
Stocks .....	19.2

As of August 31 the total cost of investments was \$232,223.90 and the market value was \$202,459.30. The market value of these holdings has increased since August 31 and the reserve for market depreciation of \$25,000.00 still covers the difference.

The August 31 statement of income and expense for the year ending August 31, 1938, is as follows in total:

Income .....	\$320,823.65
Expense .....	284,493.18
Net Gain .....	\$36,330.47
Income from METAL PROGRESS increased approximately 5 per cent this year over the prior year from .....	\$105,640.68 to \$111,769.37
but as expense increased 13.5 per cent from .....	96,204.21 to 109,236.82
the Net Gain was reduced from .....	\$ 9,436.47 to \$ 2,532.55

Book sales again show a substantial contribution to the surplus account, the net addition for the year amounting to over \$7600.00.

Income from METALS HANDBOOK sales necessarily was comparatively low this year as the supply was limited and was exhausted several months ago. The total sales of \$2541.93, however, nicely balanced expense for this 1936 edition. The new edition is well along and will be completed the early part of 1939, the expense for this year in preparing the new edition for printing amounting to a little over \$10,000.00.

The 1937 Atlantic City Convention and Exposition was another outstanding success financially, artistically and as a sales and advertising medium for exhibitors.

In addition to the Atlantic City show we had a Western Metal Exposition last March in Los Angeles which compared very favorably with the National show and it is very gratifying to report that in addition to serving a definite technical need in the growing industrial activity of the West Coast, was able to meet all expenses and show a small excess.

The membership, which is the foundation of all activities, continues to maintain the high total of members reached a year ago, in spite of reduced industrial activity and gross dues receipts of \$103,600.00 is only slightly lower than last year's total of \$105,600.00. Approximately \$41,500.00 was returned to the chapters.

In addition to the figures shown above, reports from the chapters show a total net worth of over \$68,000.00 in assets held by them. This is an increase of \$10,000.00 over last year's report and speaks very well for the healthy financial condition of the local chapters.

### CONDENSED AUDITED BALANCE SHEET AMERICAN SOCIETY for METALS

As of August 31, 1938

ASSETS	
Cash .....	\$ 70,667.89
Securities .....	209,130.00
Accounts receivable .....	8,348.08
Inventories .....	24,479.32
Other assets .....	5,758.01
Office furniture, fixtures and equipment .....	7,158.10
Deferred charges .....	16,611.78
	<hr/>
	\$342,153.18
LIABILITIES AND SURPLUS	
Accounts payable .....	\$ 2,657.09
Reserves .....	45,000.00
Deferred income .....	55,900.75
Surplus .....	238,595.34
	<hr/>
	\$342,153.18

The above from Balance Sheet prepared by Ernst & Ernst, Certified Public Accountants.

# **INCOME AND EXPENSE** **AMERICAN SOCIETY for METALS**

For the year ended August 31, 1938

## **INCOME**

### **METAL PROGRESS**

Advertising .....	\$103,894.50	
Subscriptions .....	3,164.78	
Advertisers Service .....	2,270.56	
Reprints .....	2,163.03	
Sales .....	276.50	\$111,769.37

### **MEMBERSHIPS**

Membership dues .....	\$103,601.25	
Sustaining exhibitors dues .....	4,500.00	\$108,101.25
Less apportionment of dues to chapters .....	41,476.34	66,624.91

### **1937 CONVENTION—ATLANTIC CITY**

Space rental .....	\$ 72,559.00	
Other income .....	6,158.30	78,717.30

### **WESTERN METAL SHOW—LOS ANGELES**

Space rentals .....	\$ 30,755.00	
Other income .....	3,597.40	34,352.40

Sales—Metals Handbook .....	2,541.93	
Books published and purchased for resale .....	11,127.74	
Interest and dividends .....	6,992.69	
TRANSACTIONS—bound copies, subscriptions, etc. ....	5,007.79	
Discount earned .....	1,809.88	
General reprints .....	599.82	
Membership certificates .....	533.35	
Chapters—technical service .....	311.30	
Profit on disposal of securities .....	53.75	
THE REVIEW—advertising, subscriptions, etc. ....	24.25	
Miscellaneous .....	357.17	

<b>TOTAL INCOME .....</b>		<b>\$320,823.65</b>
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## **EXPENSES**

### **As shown by schedule**

<b>METAL PROGRESS .....</b>	<b>\$109,236.82</b>	
1937 convention—Atlantic City .....	64,495.24	
Western Metal Show—Los Angeles .....	28,106.06	
Metals Handbook .....	10,099.94	
Books published and purchased for resale .....	3,492.44	
TRANSACTIONS—bound copies, subscriptions, etc. ....	22,029.25	
General reprints .....	306.64	
THE REVIEW .....	5,020.19	
General expense .....	10,504.76	
Secretary office .....	10,989.20	
Chapters .....	7,747.85	
Accounting department .....	6,541.64	
Expense of trustees .....	1,576.11	
Expense of president .....	857.36	
National committees .....	1,534.99	\$282,538.49
Provision for doubtful accounts .....	1,000.00	
Lectures, medals, etc. ....	814.10	
Books for library .....	89.07	
Emblems, pins and buttons .....	51.52	

<b>TOTAL EXPENSES .....</b>		<b>\$284,493.18</b>
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<b>NET INCOME .....</b>		<b>\$ 36,330.47</b>
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The above from Income and Expense sheet prepared by Ernst & Ernst, Certified Public Accountants.



## ANNUAL REPORT OF THE SECRETARY

October 19, 1938

The American Society for Metals, on October 1, 1938, had a total membership of 9923. Of this number 8487 or 85.6 per cent were the member classification; 617 or 6.2 per cent were sustaining members; while 763 or 7.6 per cent were juniors.

On October 1, 1937, the Society had a total membership of 10,028. This shows a decrease for the year of only 1 per cent, which, in view of the conditions of the past year, is a creditable showing.

*Publication Committee*

The Publication Committee for the year 1937-38 was made up of the following: A. A. Bates, Chairman; W. H. Bassett, Jr., A. L. Boegehold, J. L. Burns, John Chipman, Earnshaw Cook, C. N. Dawe, R. H. Hobrock, L. W. Kempf, V. N. Krivobok, O. W. McMullan, N. I. Stotz, W. H. Swanger, and Ray T. Bayless, Secretary.

Throughout the year and up to the present time the Committee has reviewed and approved 42 papers, rejected 5, and is in the process of reviewing 11 at the present time, and 1 has been withdrawn by the author.

The last meeting of the Publication Committee was held in National headquarters office in Cleveland on December 10, 1937. It was at this meeting that the subject for the symposium at the present Convention was decided upon, i.e., "Effects of Alloying Elements on the Hardenability of Medium and Low Alloy Steels (Up to 5 per cent Alloys)" as well as the appointment of a sub-committee for this symposium which was made up of the following members: A. A. Bates, Chairman; A. L. Boegehold, M. A. Grossmann, H. W. Gillett, H. W. McQuaid, N. I. Stotz, and Ray T. Bayless, Secretary.

At this meeting it was decided that the subject of the 1939 symposium would be "Precipitation Hardening (Age Hardening) of Metals" and that chairman Bates was authorized to appoint a sub-committee to make arrangements for the 1939 symposium. This he did with the following persons: A. A. Bates, Chairman, H. P. Croft, R. H. Harrington, L. W. Kempf, Howard Scott, Joseph Winlock, and Ray T. Bayless, Secretary.

On January 8 the 1938 symposium committee met and made arrangements for the symposium for this year's convention in Detroit.

On June 3 the 1939 symposium committee met and made arrangements for the symposium for the 1939 convention to be held in Chicago. All assignments of papers have been made and accepted by the selected authors and should result in a very well rounded symposium on the subject.

### *Transactions*

There have been published since the last annual meeting of the Society, four quarterly issues of TRANSACTIONS, i.e., December 1937, March, June and September 1938. The number of pages published in these four issues totals 1194 pages. There were 47 papers printed together with the discussions. A total of 42,800 TRANSACTIONS has been distributed to the membership containing a total of 12,775,800 pages.

The TRANSACTIONS published since December 1937 contain papers presented at the 1937 Convention together with others received during the year.

### *Metal Progress*

A new high in publication activity was reached by METAL PROGRESS, your monthly magazine, during the fiscal year 1938, due to an enlarged circulation and a gratifying number of advertising pages. Average circulation for the year was 11,800 copies monthly; average magazine size was 128 pages; total pages circulated therefore reached the enormous number of 18 million.

Net paid advertising for fiscal 1938 totaled 877.5 pages; for fiscal 1937 the total was 867.5. Comparison of the figures, issue by issue, shows that the early months of fiscal 1938 registered good gains over corresponding months of fiscal 1937, thus slightly more than offsetting recent losses—which by the way were very minor until June of this year. The effects of the recession which set in with the Stock Market collapse during our last annual convention were therefore not felt by METAL PROGRESS advertising for nine months. This places your magazine in a unique and enviable position among the trade and technical press, as far as advertising in 1938 is concerned. The present annual reference issue, dated October and now in your hands, is not included in the above statement, it being part of fiscal 1939's operation. It may be looked upon, however, as an indication of nearby prospects; its advertising volume is almost

exactly equal to the average of its three predecessors, 1937, 1936 and 1935.

Many favorable compliments reach the National Office as to the value of the editorial content, the mode of presentation, and the attractive covers of METAL PROGRESS. A concrete expression of the high regard the publishing industry holds for your magazine is a plaque representing "Honorable Mention to METAL PROGRESS for publishing achievement during the twelve months ending August 1, 1938," awarded in the annual competition conducted by *Industrial Marketing*, and supervised by the American Industrial Advertisers' Association. The award was made at the Association's recent convention in the words: "A first award has been made for greatest improvement in typography during the past 12 months, but it is no detraction to the winner to put on record the fact that if the terms of the award had not been specifically for improvement in the past 12 months, the first place rather than honorable mention would have gone without competition to Metal Progress, which has maintained a uniquely high standard of typographical excellence since its foundation eight years ago."

The Editorial Advisory Board for METAL PROGRESS has held quarterly meetings. A serious problem has been to avoid a complaisant feeling. In order to get a direct answer to some pertinent questions raised as to editorial content and treatment, it has authorized an entirely independent survey of "reader acceptance." Field work on this has just been completed by Market Research Corporation of New York. The entire membership was scientifically sampled so that the result represents personal interviews with a typical cross section of the membership, properly proportioned in three different ways, namely, geographically, industrially and functionally. The following may be quoted from the summary of this survey: "From personal interviews 99 per cent of the membership were found to be reading the magazine; the majority spend more than one hour's time with each issue (weighted average  $1\frac{3}{4}$  hours). In addition, 67 per cent of all the people interviewed find they get benefit from Metal Progress not derived from other sources. The style of articles, illustrations and general contents of the magazine are approved by a majority of the members. Appreciation of the magazine is further indicated by the fact that 82 per cent of the members save all or part of the copies, and 25 per cent pass them on to an average of five people."



### *Preprints*

Forty-three papers were presented before the nineteenth annual Convention held in Atlantic City last year. For this year's Convention 40 papers have been scheduled for presentation, 33 of which have been preprinted and distributed to those members requesting them. The total number of pages of preprints for 1937 was 563; the total number of preprint pages for 1938 is 792 pages. A total of 39,600 preprints have been distributed to the membership or a total of 950,400 pages.

### *Books*

Since the last Convention, two books have been published and made available to members and others, i.e., the Carburizing Symposium and the book by J. R. Vilella on "Metallographic Technique." At the present time we are preparing a treatise on forgings, both small and large. This book is by W. Naujoks and D. Fabel, and will be published about the first of the year 1939.

Three new publications for the coming year will be the result of material presented at this convention.

One will be on the papers presented in the symposium on "Hardenability of Medium and Low Alloys Steels"; the second will be the lectures presented in the educational course on "Machinability," and also the lectures presented in the education course on "Pyrometry."

### *The Review*

During the past year THE REVIEW has been published monthly with the exception of July and September. Of the ten issues which appeared from November 1937 to October 1938 three were 12 pages and seven were 8 pages.

The October issue was devoted almost entirely to the National Metal Congress and Exposition, carried 12 pages, and had a circulation of about 17,000 among the members of the co-operating societies in the Congress.

Of the total space used in the ten issues during the past year, 3787 column-inches or 82 per cent was devoted to editorial matter, and 813 column-inches or 18 per cent was devoted to advertising. Reports of chapter meetings used 1533 column-inches or 33 $\frac{1}{3}$  per cent of the total space.

*Metals Handbook Committee*

A complete report indicated the work that had been and was being covered by the sub-committees and authors of various HANDBOOK articles.

The personnel of the Handbook committee is as follows:

R. L. Dowdell, Chairman	A. O. Schaefer
J. E. Donnellan, Secretary	R. B. Schenck
R. H. Aborn	K. R. Van Horn
E. L. Bartholomew	Jerome Strauss
A. D. Beeken	H. L. Maxwell
O. E. Harder	J. H. Hall
S. L. Hoyt	C. W. Obert

The committee was able to report that excellent progress was being made on the preparation of the new edition, that it would contain approximately 120 new articles, with the prospect that 32 additional assignments that had been made to authors would be completed in time for inclusion.

The approximate length of articles in the present edition of the HANDBOOK is between four and five pages. If the new articles received should average the same, it is estimated that the new book will contain about 2000 pages.

A very comprehensive index for the new METALS HANDBOOK is in preparation.

*Educational Committee*

The Educational Committee under the chairmanship of R. L. Kenyon, with a membership of the following: E. B. Drake, W. E. Harvey, R. G. Roshong, E. C. Bain, N. L. Mochel, W. H. Eisenman, G. B. Waterhouse, and Ray T. Bayless, has studied for a period of a year or more the possibilities of Society activities in visual educational field.

In the beginning, the obvious assumption was that the moving picture method offered the greatest possibility for visual educational activities and as a result numerous persons and companies have been contacted to obtain information, data and advice as to the feasibility and methods of procedure. Sound motion pictures, slides with movie tone lecture accompanying, and combinations of these two have been carefully considered. Approximate figures have been obtained as to the cost of producing given lengths of film and slides with movie tone.

Numerous subjects have been considered as the possible starting points for an activity along these lines but after mature consideration of the many factors obtained, the Committee feels that while it has made much progress in this study it is not in a position at this time to make a definite recommendation as to the best procedure to be taken, if any, by the Society in producing one or a series of educational lectures by any one of the above methods.

### *1938 Convention Educational Program*

The Educational Committee feels rather proud of the program that it has arranged for this Metal Congress, i.e., a five lecture series on the Machinability of Metals which is being presented on five consecutive afternoons at 4:30 P.M. as follows:

1. Physics of Cutting—by Hans Ernst, Cincinnati Milling Machine Co.
2. Machinability of Ingot Iron, Wrought Iron, S.A.E. Steels and Stainless Steels—by H. B. Knowlton, International Harvester Co.
3. Some Factors Affecting the Machinability of Cast Steel, Cast Iron, and Malleable Iron—by J. W. Bolton, Lunkenheimer Co.
4. Machinability of Tool Steels—by A. H. d'Arcambal and W. E. Bancroft, Pratt & Whitney Co.
5. Machining Nonferrous Materials (Cast and Wrought)—by H. P. Croft, Chase Brass and Copper Co.

The evening lecture series of three lectures by Dr. R. B. Sosman on the subject of Pyrometry has been an outstanding contribution to the subject and has been of real benefit to those who have heard them.

### *Metal Congress and Exposition*

If it were not that this annual report, which is published in full in the December TRANSACTIONS, should be a complete record of the activities of the year, it would be carrying "coals to New Castle" to speak of the success that is attending the present National Metal Congress and the National Metal Exposition.

You have had an opportunity to attend the sessions—you have observed how well they are attended, how capably the speakers are presenting their investigations, and what a splendid spirit of fellowship exists among the membership.

As you have visited the National Metal Exposition you could not have failed but to observe and realize that it is the largest Metal Exposition ever held; that it is approximately 20 per cent larger than



the one in Atlantic City last year; and judging from the interest displayed in the exhibits as well as the ability of the manufacturers in adapting their splendid displays to the limitations of the building, you will have reached a sound conclusion when you characterize the 1938 Metal Show—one of the best.

### *Western Metal Congress and Exposition*

Some 18 national technical societies having divisions and chapters on the Pacific coast co-operated with the A.S.M. in staging the Western Metal Congress and the Western Metal Exposition in Los Angeles, March 21 to 25, 1938.

Quite an outstanding technical program was prepared around the theme, "Metals in Industry," and the 49 papers contributed to that Congress as well as the independent sessions held by the American Welding Society combined to afford an excellent technical program, especially adapted to the industries of the West.

Some 185 exhibitors participated in the Western Metal Exposition, which occupied approximately 35,000 square feet and was attended by 36,000 people.

Those in attendance from the technical angle as well as those in attendance at the exhibit were equally enthusiastic of the splendid results accomplished and were sincere in their enthusiasm that the event should be repeated in 1940.

The reports which have been briefly presented by the President, the Treasurer and the Secretary, are mighty like a symphony of success, but not an unfinished symphony. It is well that we should let the past be the past, but it is also appropriate that we might at this moment crystalgaze as to what the coming year has in prospect for the members of the Society.

We well know that the activities planned by the chapters for the coming year not only in the programs arranged for their regular meetings but in the splendid educational activities that have been proposed, and some of which are now in progress, will combine to the goal that the chapters will be of greater service to their members and to the industries located under their influence.

METAL PROGRESS, which has been under the able editorship of Ernest E. Thum for the past eight years, will continue under his supervision, and you may expect a continuance of the well-prepared and well-edited articles dealing with the production and fabrication of both the ferrous and nonferrous metals—arranged with the same

high-grade typography which won a national award this year—together with appropriate data sheets; and another outstanding convention issue—all of which will be accompanied by a galaxy of advertising pages which have become recognized as an authoritative catalog in the industry.

You may anticipate that *TRANSACTIONS*, which has been under the efficient editorship of Ray T. Bayless for the past eighteen years, will continue to maintain the high standard of technical literature that it has in the past. The papers which are being presented at this convention, which form the backbone of material for the quarterly *TRANSACTIONS*, have evidenced that in scope, character and value they will again produce a volume of metallurgical literature equalled no place else on the globe.

You members will receive early during the coming year the 1939 edition of the Metals Handbook whose 2000 pages will have been prepared with the splendid and helpful co-operation of the Metals Handbook Committee and under the able direction of J. Edward Donnellan, who has served this committee for fifteen years. The Handbook will present a metallurgical achievement in which you may well take pride. The new edition will again be exchanged free of charge to all members of the Society in good standing.

The National Metal Congress and the National Metal Exposition, scheduled to be held in the city of Chicago next year, will undoubtedly fulfill your fondest expectations, and because of the effort and energy that will be placed into their successful completion, the useful purposes will be increased and expanded.

You may anticipate that the work of the Educational and Publication Committees will bring to you greater service and additional helpful metallurgical knowledge and assistance.

You may expect the Finance Committee to give its serious attention to financial affairs and endeavor to guide your Society safely through the shoals of any economic disturbances which may make their appearance above the horizon.

And finally, you may look for certain and be assured that you will have from the officers and members of the Board of Trustees of the A.S.M. as well as from the staff in national headquarters, a super-application to the welfare of the Society in order that mature judgment and experience and conscientious attention will provide a year of activity and accomplishments, the results of which will be apparent to all, and a source of genuine satisfaction to the membership.

**ELECTION OF OFFICERS**

GEORGE B. WATERHOUSE, *President*

Complying with the Constitution, I appointed as of March 15, 1938, the following Nominating Committee, selected from the list of suggested candidates received prior to March 1, 1938:

Emil Gathmann (chairman), Baltimore Chapter  
Adolph O. Schaefer, Philadelphia Chapter  
E. O. Dixon, Milwaukee Chapter  
John W. Bolton, Cincinnati Chapter  
M. W. Carruthers, Rhode Island Chapter  
L. C. Conradi, Southern Tier Chapter  
A. G. Zima, Los Angeles Chapter

This Committee met at State College, Pennsylvania, on May 19, 1938, and made the following nominations:

President—W. P. Woodside, Climax Molybdenum Co., Detroit.  
(1 year)  
Vice President—J. P. Gill, Vanadium Alloys Steel Co., Latrobe, Pa.  
(1 year)  
Secretary—William H. Eisenman, Cleveland.  
(2 years)  
Trustee—D. S. Clark, California Institute of Technology, Pasadena, Calif.  
(2 years)  
Trustee—Francis B. Foley, Midvale Co., Philadelphia.  
(2 years)

A report of these nominations duly appeared in *THE REVIEW* for May 1938.

I have been informed by the Secretary that no additional nominations were received prior to July 15, 1938, for any of the vacancies occurring on the Board of Trustees. Consequently the nominations were closed. I shall now call upon the Secretary to carry out the provisions of the Constitution in respect to the election of National Officers.

W. H. EISENMAN: Conforming with the requirements of the constitution of the Society, I hereby cast the unanimous vote of all members for the election of the aforementioned candidates, who were nominated on May 19, 1938.

PRESIDENT WATERHOUSE: The provisions of the Constitution having been complied with, I hereby declare the candidates heretofore named to be duly and unanimously elected to the several specified offices, the terms of each office just elected beginning on the day following the close of this annual meeting.



## THE ANNUAL BANQUET

On Thursday evening, October 20, 1938, the American Society for Metals was host to more than a thousand members and guests at its annual banquet held in the ballroom and banquet room of the Hotel Statler, Detroit. Many executives representing important steel producing and steel consuming companies were in attendance. Seated at the speakers' table were the following: B. B. Beckwith, metallurgical department, Chrysler Corp., and secretary-treasurer of the Detroit Chapter; S. L. Hoyt, director of research, A. O. Smith Corp., and trustee-elect of A.S.M.; R. L. Wilson, assistant metallurgist, Climax Molybdenum Co., and trustee, A.S.M.; Axel Hultgren, professor of metallurgy, Stockholm; Bradley Stoughton, dean of engineering, Lehigh University, and treasurer of the A.S.M.; Ralph W. E. Leiter, research metallurgist, Edward G. Budd Mfg. Co., and 1938 Henry Marion Howe Medalist; Joseph Winlock, research metallurgist, Edward G. Budd Mfg. Co., and 1938 Henry Marion Howe Medalist; A. L. Boegehold, research laboratories, General Motors Corp., the 1938 Edward DeMille Campbell Memorial Lecturer and chairman of the Detroit Chapter; J. M. Schlendorf, assistant vice-president in charge of sales, Republic Steel Corp.; C. E. Wilson, vice-president and general assistant to president, Chrysler Corp.; Edgar C. Bain, assistant to vice-president, U. S. Steel Corp., and past president of the Society; Benjamin F. Fairless, president, U. S. Steel Corp., and principal speaker of the evening; George B. Waterhouse, head of department of metallurgy, Massachusetts Institute of Technology, and president of A.S.M.; W. P. Woodside, vice-president, Climax Molybdenum Co., and a founder member and president-elect of the A.S.M.; Frederick J. Griffiths, retired steel executive; Dr. Albert Sauveur, Professor Emeritus, Harvard University; H. W. McQuaid, metallurgist, Republic Steel Corp., and 1938 recipient of the Sauveur Achievement Award; J. P. Gill, metallurgist, Vanadium-Alloys Steel Co., and vice president-elect of A.S.M.; O. W. Ellis, director, Ontario Research Foundation and trustee of the A.S.M.; C. H. Colvin, Jr., president, Rotary Electric Steel Corp.; J. M. Watson, past president of the A.S.M.; H. A. Anderson, metallurgical engineer, Western Electric Co., and trustee of the A.S.M.; F. B. Foley, superintendent of research, Midvale Company, and trustee-elect of the A.S.M.; D. S. Clark, instructor of mechanical engineering,

California Institute of Technology, and trustee-elect of the A.S.M.; W. H. Eisenman, secretary of the American Society for Metals.

President Waterhouse presided at this meeting and expressed the appreciation of the trustees and the membership of the American Society for Metals to the Institute of Metals, and the Iron and Steel Divisions of the American Institute of Mining and Metallurgical Engineers, the American Welding Society, the Wire Association and the American Society of Mechanical Engineers for their splendid co-operation in making this present convention an outstanding success.

Each of those seated at the speakers' table were introduced to the assembled audience. Several awards were then made in the following order: To Dr. E. C. Bain was presented a past president's medal in recognition of the splendid services he had rendered the Society in his various capacities on the Board of Trustees.

A certificate of appreciation was awarded to A. L. Boegehold, the 1938 Campbell Memorial lecturer, as a token of our esteem for the splendid lecture he presented. To Joseph Winlock and Ralph W. E. Leiter was presented the Henry Marion Howe medal and certificate for their paper entitled "Some Factors Affecting the Plastic Deformation of Sheet and Strip Steel and Their Relation to the Deep Drawing Properties," which was adjudged the one of highest merit published in the *TRANSACTIONS* of the Society during the preceding year. To H. W. McQuaid was presented the Albert Sauveur Achievement Award and in presenting the candidate for this award, Dr. Bain made the following citation which had justified the selection of Mr. McQuaid as the candidate of the award for 1938.

"The exceedingly pleasant assignment has been given me of presenting to you, Professor Sauveur, on behalf of the Committee of Past Presidents, the candidate whom we have selected to receive from your hands the Albert Sauveur Achievement Award. The Past Presidents have deliberated well and our candidate is designated in this citation:

'In recognition of his pioneer studies upon the grain-size and microscopic features of carburized steel, and particularly in recognition of the resulting stimulation of research upon grain-size and hardenability of steel, the Albert Sauveur Award in 1938 is conferred upon Harry W. McQuaid.'

"Mr. McQuaid can scarcely remember a time when, as a boy, he

did not feel confident that he would be an engineer. At the age of 10 years, he undertook an ambitious, though perhaps not quite perfect, survey of his father's farm near Englewood, New Jersey, where he later attended high school. Years later, at Stevens Institute of Technology, he learned that more mature engineers also had their difficulties with the error in closure of the transverse.

"After receiving the bachelor's degree in Mechanical Engineering at Stevens in 1913 he soon found himself taking care of the power plant at the United Piece Dye Works at Lodi, New Jersey, and soon after engaged himself with problems in the application of heat: 'soldering irons to soaking pits.' McQuaid laconically explains this activity.

"In the Autumn of 1915, he became night foreman of the heat-treating department at Timken in Canton, Ohio, standing guard against the old enemies—soft spots and cracks—in case-hardened parts. Here his experience soon widened as he was successively electrician on the electric furnace installation, and later helper. In 1918 he went to the metallurgical laboratory where much of his important work, mentioned in the citation, was done. Systematically following individual heats as to hardening characteristics and microstructure he made the observations which resulted in his now familiar coarse and fine, normal and abnormal basis of classification. From McQuaid's reminiscences of those days one learns that they were exciting ones, of well-filled hours under the inspiring leadership of M. T. Lothrop, to whom he acknowledges a great indebtedness.

"In 1920 Erik Ehn joined the laboratory staff and one may infer from McQuaid's account that the partnership of McQuaid and Ehn was happily one of mutually complimentary abilities. By 1922 the concept of microscopic differences in slowly-cooled carburized specimens, as between heats of inferior and heats of superior hardening capacity was well matured and in use; accordingly a joint paper was read in February before the A.I.M.E., a patent application relating to the test having been made in 1921. The paper drew forth considerable discussion and in a short time metallographists in all quarters were examining carburized specimens.

"In 1926 he was transferred to the Timken Detroit Axle Company in Detroit, where he remained until 1933, when he joined the technical staff of Republic Steel Corporation as metallurgist. In 1935 he was chosen to deliver the tenth Campbell Memorial Lecture; his subject—"The Importance of Aluminum Additions in Modern Com-



mercial Steels." Other McQuaid publications include a contribution to the 1934 Grain-Size Symposium, a paper with O. W. McMullan, as well as several articles on case carburizing and the effect of aluminum in steel. He was active in establishing the single direct quench as a suitable practice for fine steels in high grade work, in introducing a generally higher hardness in automotive parts, and in the use of higher carbon in high speed steel tools.

"In the grain size work, for which he is perhaps best known, McQuaid collaborated with his steel producing neighbors in Canton and Massillon. While at first McQuaid and Ehn found a simple designation coarse and fine adequate for grain-size, later refinements were introduced. We understand that at United Alloy Steel Company a 10-step series of standard grain-size photomicrographs was prepared in 1924 while in 1926 a 9-step series with transition and core zones added appeared from Central Alloy Steel Company. At any rate by 1928 the Timken series, now also the A.S.T.M. standard, of 8-steps in a geometrical progression of grain-sizes was prepared and is now in general use.

"If anyone were to say that new ideas are immediately accepted in American metallurgical circles without critical discussion he would be judged to be seriously ill-informed. Nor was this fascinating story of structural normality an exception. The basic central theme of the story was confirmed by many investigators while the significance and explanation of the observations became a subject of a plentiful amount of good healthy scientific controversy. Many of the brilliant researches prompted, at least in part, by the McQuaid publications were discussed before this Society, an example of the fostering of unhampered expression of view in which we may take pride. To mention only a few, the names of Brophy, Davenport and his Kearny associates, Epstein, Graham, Grossmann, Herty, Houdremont, Luerksen, Mehl, Nead, Rawdon, Scott, Shane, Sanders and White, come to mind. Who can say how much our present clear understanding of grain-size and hardenability was hastened by the work of Harry McQuaid, or how far we should fall short of this status had McQuaid and Ehn not made their observations!

"The Albert Sauveur Award is intended to reward those pioneer achievements, particularly, which have outstanding subsequent effect upon the thought and trends of study in the field. One might say, in short, that it is for those who really start something. We have seen McQuaid-Ehn tests employed by the tens of thousands and

observed that masterful researches have been directed to the subjects revealed in his early contributions.

"It is then an honor and a pleasure to present for the 1938 Albert Sauveur Achievement Award, Harry W. McQuaid."

The award was then made personally by Dr. Albert Sauveur.

Following this presentation the guest of honor and speaker of the evening, Benjamin F. Fairless, president of the United States Steel Corporation, gave his outstanding address which is printed in full herewith.

### **The Scientific Approach To Our Problems**

BY BENJAMIN F. FAIRLESS, PRESIDENT,  
UNITED STATES STEEL CORPORATION

*Presented at the Annual Banquet of the American Society for Metals  
October 20, 1938, Detroit*

"An occasion such as this is one of significance to all who are interested in the production and use of metals. Each meeting of the American Society for Metals seems to constitute a post graduate course in metallurgy, and fortunate indeed are those who are privileged to attend its sessions. Along with your associates in the National Metal Congress you are making available, year by year, a fund of new and useful information—information which contributes in an important way to the progressive development of industry. It is evident that inspiration and new ideas are to be had here for less than the asking. Moreover the convenient opportunity for valuable personal contacts with men of common purpose, leaders in the science and technology of metals, may be readily appraised as one of the major assets of this great fraternity. It is with a feeling of pride in your accomplishments, sympathy with your declared aims and purposes, and an earnest wish for the continued success of the Society, that I acknowledge the pleasure you have afforded me by providing a place at your table tonight. The honor of the occasion, as well as the privilege of enjoying your company, is deeply appreciated.

"Happily for all of us at this time, we are able to watch the curve of business activity in the United States move patiently upward. The ascent is slow, and not without irregularities, but at least the slope is in the right direction. In comparison with the conditions prevailing during the first half of the year 1938, the current per-

formance of industry is a cause for some gratification, and the outlook for the nearby future holds promise of further improvement.

"Reports emanating from various sources permit one to harbor a measure of optimism. For example, there is evidence that certain classes of inventories have been adjusted so that they need not stand in the way of a direct reflection of customer demand upon producing facilities. Car loadings have been increasing, recently making a new peak for the year. Automobile output has risen by a substantial percentage as the automotive industry enters its new season. Various, though not all industries are contributing reports and forecasts which offer words of cheer. The general situation is described in a statement which appears in the October issue of *The Federal Reserve Bulletin*, as follows—"The increased activity that has occurred since early summer has been reflected in a sharp rise in the Board's seasonally adjusted index of industrial production." Thus while we realize that comparisons with the year 1937, in many lines of business, still do not yield figures which warm the heart or meet one's wishes, the forward movement which has taken place in these latter months of 1938 is encouraging. We are prepared to move along from our present position, in the high hope that the integrated wisdom and judgment and efforts of the people in this country will prove equal to the task of coping with the social, political, and economic problems which affect our progress.

"The situation of industry surrounded by a group of perplexing problems does not offer a new experience to any of us. We have come to look upon this state of affairs as an inevitable accompaniment of our daily activity. Within the past nine years, however, during which wide fluctuations from normal have occurred, and the business cycle, or whatever is called the business cycle, has been performing some remarkable antics, the complexity and variety of our problems seem to have increased. The executive today, in coming to an important decision, consults not only those agencies which helped him in years gone by, but also many others peculiar to our recent times. Industrial organizations, with the aid of experts skilled in matters now looming as large factors in the equations of business, are continually bending their efforts toward finding the correct answers. Probability and chance are favorite branches of mathematics, these days.

"In the maze of this diversified activity it is refreshing to pause for a moment, look toward the metallurgical sector, and consider how



well you gentlemen and your companions have come to the aid of the metals industry, in both its producing and consuming branches. Your special domain is certainly not quiet, nor is it devoid of unsolved problems. Experience indicates that it never will be so. But it is at least in comparatively orderly condition. It is being explored and cultivated systematically, and from it there issue developments which count heavily in the conduct of business. Within the period of a relatively few years, substantial results have been achieved as the outgrowth of a policy which seeks—'to promote the arts and sciences connected with either the manufacture or treatment of metals, or both'—as your By-Laws phrase it.

"Speaking more particularly of the steel industry, because it serves well to illustrate the case, we see the many notable changes that have taken place in consequence of the impact of chemistry, engineering, and physical metallurgy upon its problems. These influences have been felt for approximately forty years, but in a more important and significant manner only since the close of the World War. What was formerly the art of steel making or of heat treating has moved rapidly along the road toward becoming a science. A vast portion of the hitherto unknown has been illuminated and explained by technological effort. Methods and practices which insure better processes and superior products are now available to the steel industry and to its customers. New grades of steel have been brought forth in profusion.

"Such matters as grain size, aging, creep strength, microstructure, controlled transformation, and a host of others, which only a few years ago meant little or nothing to the producers and users of metals, are very real considerations in the every day transactions of the trade. It is hardly necessary, at this meeting of technologists tonight, to elaborate further or to describe the substantial benefits which have followed in the wake of exact knowledge. You men have been in the thick of the development and are in daily contact with the results. You know, as well as any and better than most, how much would have been lost to the metals industry had it not adopted the scientific method of attack upon its problems.

"This realization should bring a sense of satisfaction to you, as it does to us, because together we can feel that in these difficult times, at least the technological aspects of our business are being handled in a rational manner. There is nothing more stimulating than an honest effort of the intellect, consistently directed toward

overcoming difficulties and uncovering the ways and means of progressive improvement. Metallurgy and engineering have kept their houses in order with the prosecution of just such effort, despite the hurly-burly of the past decade. Register a vote for applied science!

"A little while ago, an allusion was made to the work of the metals technologist as being unfinished. There are many reasons why this should be so. In the first place, a forward step in one line of activity almost inevitably paves the way for advancement in many collateral and dependent lines. Any marked improvement in blast furnace or open-hearth procedure, for example, affords grounds for the adjustment of a dozen subsequent processes, reaching even into fabricating and finishing operations. Following the usual course, a development in the metals industry, although it may start as a point, usually spreads out fanwise before it is finished. All of you know from experience that the work of the metallurgist is not at an end with the genesis of a new grade of alloy steel or the birth of a non-ferrous alloy. So it appears that there will always be plenty of work to do. The momentum gathered by the stream of technological effort and invention in recent decades is proof against sudden stoppage or diversion.

"Among the other reasons which insure a continuing place for technical work in the metals industry, only one will be singled out for discussion. It is comprehended within the meaning of the term, competition. There is, first of all, competition between and among the concerns which make up the steel industry. This is normal and stimulating. A wide-awake neighbor is usually an asset. In the second place, you are aware of a keen rivalry between ferrous and nonferrous metals in certain fields of application. Displacements here and encroachments there are mutual—and add zest to the game. But to the metals industry as a whole, the third and perhaps most interesting form of competition is provided by the sponsors of non-metallic materials. Paper, wood, glass, moulded plastics, and cement are continually looking for opportunities to replace the metals, just as the metals are making an effort to fit themselves into the places occupied by the group just mentioned. All of this can mean but one thing, that every actor in the arena of business is going to be alert to the last degree, or yield ground to his progressive competitors. It is essential that each and every industry make use of all the aid that science can render, to carry its products through the contest. In the long run, the prizes will be awarded on the basis of merit—

and the consuming public will share in the benefits. It is our intention that with your assistance, the metals shall not lose ground.

"The steel industry, along with others, has given evidence of its faith in the possibility of finding workable solutions to the perplexing problems lying beyond the field of technology. Notwithstanding the presence of other unknowns, it has taken to heart the developments and accomplishments of the metallurgist, the engineer, the chemist, and the physicist, and has revamped much of its physical equipment to take advantage of their findings. The cost has been substantial. Within the past five years the steel industry has spent approximately eight hundred and fifty million dollars for improvements. Old methods and machinery have been discarded as obsolete or inefficient, in the light of what has been learned in recent years. Quality of product, service to customers, and economies in operation have been important factors in this movement. Some critics may say that the steel industry has been overbuilt. Considerable discussion, for example, revolves around the case of the continuous wide strip mill. With respect to certain capacities, and viewed from the standpoint of today's demands, there are doubtless additional problems to be solved. But how, may we ask, could any important producer in the steel business close his eyes to the current trend, refuse to modernize, and hope to live in competitive markets with a less adaptable product? If we are prepared to admit that the existent uses for steel cannot be expanded, and that no new applications can be developed, then we should be prepared to admit that the problem presents more than the usual elements of difficulty. A complete answer is not to be expected without a reasonable period of trial, and particularly during a time of abnormally low demand.

"It would appear that from the standpoint of technical development and physical equipment, the steel industry is in readiness to move forward. It has prepared itself to take care of the needs of its customers and meet the demands of the consuming trade. But all of the factors which affect an industry cannot be as readily handled from within, as can its own technology. We are not independent of outside influences. There are numerous problems affecting companion industries, as well as certain broad social and economic considerations, upon which all of us would be glad to have as definite word as you specialists have given your companies on the subjects peculiar to your respective lines of endeavor.

"It almost goes without saying that the steel industry, or for



that matter any large commercial enterprise, cannot make consistent headway except under auspices which provide for the economic health of the entire business structure. Pathological conditions in any section are felt throughout the whole. All of us who are interested in the production and sale of metals are concerned with the welfare of our consuming trade. As long as agriculture is beset with serious irregularities and difficulties, an important market for metal products is subject to uncertainty. There is a direct as well as an indirect effect upon purchasing power. In like manner, the situation of the public utilities, the railroads, and some branches of the mining industry, if disquieting to them, is also disquieting to their important suppliers.

"There is no secret about the plight of a number of our American industries—their troubles have been in the newspapers, the legislative halls, and the minds of the people for a period of years. But they still represent problems to themselves as well as to their companion industries. Such measures as have been brought to bear upon their cases have not been effective in restoring them to health. In this connection may not one be permitted to call attention once again to the value of the results obtained through the scientific study of all problems by dispassionate and experienced research workers, in the hope that here too their findings may be given due weight in prescribing a course of treatment.

"Beyond the scope of any one industry, but pervading and affecting all, are a number of fundamental problems which are engaging attention from every angle. In the interest of further sketching the picture we have been developing here tonight, a few of these will be mentioned for your consideration.

"All industry would be less perplexed if it could know, with reasonable certainty, whether the social well-being, as well as the economic health of large masses of people could be better served by a thirty, thirty-six, or forty hour working week. If that range of figures is too narrow, by what guiding principle shall we arrive at the one precise point, if any, where the division of man's time between work and leisure is at an optimum?

"In another direction, it would be gratifying to know, if possible, how the gross income of a business should be equitably apportioned among the different interested elements, particularly with respect to the workers, the stockholders, and the tax collector. How many of the factors can be fixed arbitrarily without doing violence

to the others, or to the business structure as a whole?

"Unemployment has been one of the most harrowing subjects before the American public for a period of eight or nine years. The manufacturing industries have been importuned to provide employment for the idle, and many persons hold the belief that practically all of the unemployed normally belong in those industries. In this connection it is interesting to note that the steel industry, during the busy months of 1937, employed a larger number of workers than it did in 1929. Nevertheless, all of us are interested in finding a solution to the unemployment problem, because as long as it persists, purchasing power in the aggregate is diminished, and all branches of industry are affected.

"There has been considerable public reference to size as a factor in industry. Questions have been raised as to the relative merits of large and of small units. It would be useful to know whether there is a standard or rule by which one might determine, on a factual basis, the approximate vertical and horizontal dimensions at which a significant change in category takes place. What actually determines whether a business enterprise is good or bad, from the standpoint of the public welfare?

"You will recognize, in the foregoing, many elements which are susceptible to fact finding and cold analysis. There are some, perhaps, which are not, but which will remain as objects of continual crusading. There is little doubt, however, that taken as a whole they comprehend some of our deepest concerns with respect to the future course of industry—and of organized society. In breaking them down into their component parts you will find the elements of many burning questions which have long been revolved in the minds of men, never more earnestly than during the past decade. They have been variously the substance of political issues, partisan conflicts, and agitation by pressure blocs. Now in the face of our unanimous desire to move forward on a firm and substantial foundation, is it not time to separate out those matters which can be treated by the methods of scientific research and handle them so, leaving only the less comprehensible for honest debate?"

Statement of the ownership, management, circulation, etc., required by the acts of congress of August 24, 1912, and March 3, 1933, of TRANSACTIONS of American Society for Metals, published quarterly at Cleveland, Ohio, for October 1, 1938, State of Ohio, county of Cuyahoga, ss. Before me, a notary public, in and for the State and county aforesaid, personally appeared Ray T. Bayless, who, having been duly sworn according to law, deposes and says that he is the editor of the TRANSACTIONS of the American Society for Metals, and that the following is, to the best of his knowledge and belief, a true statement of the ownership, management, etc., of the aforesaid publication for the date shown in the above caption, required by the Act of August 24, 1912, as amended by the Act of March 3, 1933, embodied in section 537, Postal Laws and Regulations to wit:

1.—That the names and addresses of the publisher, editor, managing editor, and business managers are: Publisher, American Society for Metals, 7016 Euclid Ave., Cleveland, O.; Editor and Managing Editor, Ray T. Bayless, 7016 Euclid Ave., Cleveland, O.; Business Manager, W. H. Eisenman, 7016 Euclid Ave., Cleveland, O.

2.—That the owner is: The American Society for Metals, 7016 Euclid Ave., Cleveland, Ohio, which is an educational institution, the officers being, President, G. B. Waterhouse; Vice President, W. P. Woodside; Treasurer, Bradley Stoughton; Secretary, W. H. Eisenman; Trustees: E. C. Bain, O. W. Ellis, R. L. Wilson, J. P. Gill, H. Anderson. All officers as above, 7016 Euclid Ave., Cleveland, Ohio.

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4.—That the two paragraphs next above, giving the names of the owners, stockholders, and security holders, if any, contain not only the list of stockholders and security holders as they appear upon the books of the company but also, in cases where the stockholder or security holder appears upon the books of the company as trustee or in any other fiduciary relation, the name of the person or corporation for whom such trustee is acting, is given; also that the said two paragraphs contain statements embracing affiant's full knowledge and belief as to the circumstances and conditions under which stockholders and security holders who do not appear upon the books of the company as trustees, hold stock and securities in a capacity other than that of a bona fide owner; and this affiant has no reason to believe that any other person, association, or corporation has any interest direct or indirect in the said stock, bonds, or other securities than as so stated by him. Ray T. Bayless, managing editor, sworn to and subscribed before me this 1st day of October, 1938.

(Seal) Arthur T. Wehrle, notary public.

(My commission expires January 20, 1941.)



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